A STUDY OF THE EFFECTS OF STRESS ON STRAIN AGING IN VANADIUM AND TANTALUM

By

LINDA PRISCILLA BECKERMAN

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"By our calculations - thinking of nothing else - by our desires - abandoning every other hope - by our efforts - renouncing all bodily comfort - we gained entry into this new world. So it seemed to us. But we learned later that if we were able to approach Mount Analogue, it was because the invisible doors of that invisible country had been opened for us by those who guard them ... Those who see us even though we cannot see them opened the door for us, answering our puerile calculations, our unsteady desires, and our awkward efforts with a generous welcome."

Rene Daumal

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By

Linda Priscilla Beckerman

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Strain aging under constant stress was investigated in the Snoek ordering region by means of yield point return tests. Vanadium specimens containing 132 ppm oxygen were aged in situ after prestraining 2.8% and 9% at rates of $6.7 \times 10^{-5} s^{-1}$, $8.9 \times 10^{-5} s^{-1}$, and $8.9 \times 10^{-4} s^{-1}$. Tests were performed for various aging times at temperatures between 338 K and 403 K, with aging stress levels from 0.27 to $0.98\sigma_f$, where σ_f is the prestrain flow stress.

The kinetics of Snoek strain aging were found to be independent of aging stress at 353 and 363 K and occurred at a rate 10 times faster than predicted by theory at 338 K. It is postulated that the interstitial atoms which are primarily responsible

for the yield point return reorder at a more rapid rate when they are in the high intensity strain fields close to a dislocation.

The maximum yield point return due to Snoek ordering was found to be independent of temperature between 353 and 373 K, and decreased when the temperature was increased to 393 and 403 K. The reduction in the maximum yield point return at the higher temperatures was attributed to the occurrence of Snoek dynamic strain aging during prestraining and reloading.

A sharp maximum in the magnitude of the yield point return was observed at $92\%\sigma_f$ at 353, 363, and 373 K after prestraining 2.8 or 9% at a rate of $6.7 \times 10^{-5} s^{-1}$ and aging for 35 or 120 minutes. A maximum yield point return was obtained at $87.5\sigma_f$ after prestraining at a rate of $8.9 \times 10^{-4} s^{-1}$. The conditions for obtaining a maximum yield point return were found to depend on the strain rate during aging. While aging at a stress level corresponding to the maximum yield return, the specimens deformed at a rate only 2 1/2 orders of magnitude slower than the prestrain strain rate. For aging stress levels above that corresponding to the maximum yield point return, the aging strain rate approached the prestrain strain rate as the aging stress approached the prestrain flow stress. It is postulated that,in this region, Snoek dynamic strain aging occurs during aging and that the

waiting time at a barrier decreases as the prestrain flow stress is approached. A decreasing waiting time results in smaller and smaller yield point returns. It is also postulated that the magnitude of the yield point return decreases for aging stress levels less than the maximum due to aging of a relaxed dislocation structure.

Strain aging under constant stress was investigated in the Cottrell strain aging region by means of yield point return tests. Tantalum specimens containing 52 ppm oxygen were aged in situ after prestraining 9% at a rate of $6.7 \times 10^{-5} s^{-1}$. Tests were performed for various aging times between 368 K and 388 K at aging stress levels of 20%, 50%, and $85\% \sigma_f$.

The activation energy of Cottrell strain aging was found to be independent of stress.

CHAPTER I
INTRODUCTION

Strain aging is an important aspect of the mechanical behavior in many metal systems. A number of mechanical properties associated with fatigue and creep loading of BCC metals are known to be affected by strain aging. Peaks in curves of fatigue life or fatigue strength versus temperature, obtained using low carbon steel, have been observed in the temperature and strain rate ranges where dynamic strain aging occurs [1,2]. Similarly, improvements in the fatigue properties of niobium, containing oxygen in solid solution, have been attributed to strain aging [3]. In addition, strain aging has been observed to reduce creep rates, reduce rupture life, and increase creep strength in niobium [4,5]. On the other hand, in a study of the interaction of fatigue and creep in a tantalum base alloy, the greatest acceleration in the creep rate was observed upon raising the temperature into the dynamic strain aging range [6]. In all of these cases, strain aging was occurring while the material was subjected to an applied stress.

Aging under stress is known to enhance the strain aging process. This effect is commonly observed

when strain aging is monitored by observing the return of the yield point. When tensile specimens are aged under an applied stress, a greater yield point return is obtained than when they are aged in a stress-free condition.

While there have been many extensive investigations of strain aging, very little work has been devoted to determining the effects of stress on strain aging. A review of the literature concerning this area of strain aging revealed that several areas require a more thorough investigation. The enhancement of the yield point return when aging is performed under stress is well documented; however, there is uncertainty as to whether an increase in the kinetics is responsible, resulting in the observation of a greater yield point return in a given interval of time, or some other mechanism, such as aging of the dislocations in a bowed-out configuration, resulting in a greater unpinning stress. In addition, the conditions under which the maximum yield point return can be observed have not been clearly defined.

Previous research in these areas has suffered from the disadvantage of not being able to maintain a constant stress level during aging, particularly at stress levels near the prestrain flow stress. In the

present study this difficulty was eliminated through the use of an electronic load-maintaining device developed for the Instron machine that allowed the stress level to be kept effectively constant during aging even at stress levels near the prestrain flow stress.

Snoek strain aging is particularly convenient for this kind of study since the mechanism believed responsible for the observed effects is simple in nature. In the Cottrell region of strain aging, the theories developed to explain the extent and kinetics of the strain aging response do so successfully only at the early stages of the process. At later times the process is complicated by saturation and precipitation. In contrast, only one mechanism occurs during Snoek strain aging, and the theory developed to explain it is not restricted to the initial portion of the process.

In order fully to understand the effects of aging stress on Snoek strain aging, several general issues related to it need to be resolved. In many cases, a discrepancy exists between theory and experimental measurements. Two areas of concern are the temperature dependence of the maximum yield point return due to Snoek aging, and the kinetics of the strain aging process. These two areas, plus those previously mentioned, constitute the focus of attention in this investigation.

The materials used were the BCC metals, vanadium and tantalum. These metals are currently under serious consideration for usage in the first containment walls of nuclear fusion reactors [7,8]. Vanadium and tantalum are also ideal BCC metals to use in a strain aging study since the solubility limits of interstitial atoms, such as oxygen, are very high. This allows one to observe the strain aging process without the complications of additional processes, such as precipitation, as may occur during strain aging of iron or steel.

The results obtained in this investigation by aging vanadium and tantalum under constant stress show several significant factors not previously recognized about the effects of the aging stress level on the strain aging kinetics and the magnitude of the yield point return.

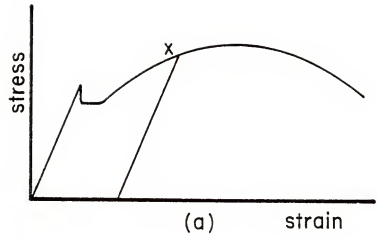
CHAPTER II
STRAIN AGING PRIMER

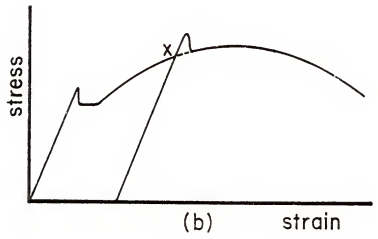
2.1 Definition

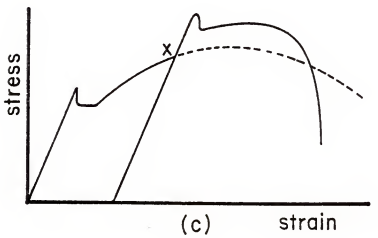
Strain aging is normally defined as a strengthening or hardening due to aging after plastic deformation. This can be explained by referring to tensile stress-strain diagrams of a BCC metal such as iron containing interstitial carbon atoms in solid solution. If, as shown in Figure 1a, one plastically deforms a specimen to point x and unloads, then upon immediately reloading, the specimen will plastically deform at the prestrain stress level point of x. If, upon unloading, the specimen is allowed to age, then upon reloading a higher stress level than that at the end of the prestrain is needed to initiate yielding (Figure 1b). This is what is meant by strengthening due to aging after plastic deformation. If the specimen is permitted to age for a long enough time, then upon reloading, the entire level of the stress-strain curve may be raised over that of an unaged specimen (Figure 1c). This is referred to as permanent hardening and may be accompanied by a lowered ductility.

Figure 1.

Schematic representation showing the effect of strain aging on stress-strain curves.
(a) No aging period between unloading and reloading.
(b) Short aging period between unloading and reloading.
(c) Long aging period between unloading and reloading.


stress
x
(a)
strain


stress
x
(b)
strain


stress
x
(c)
strain

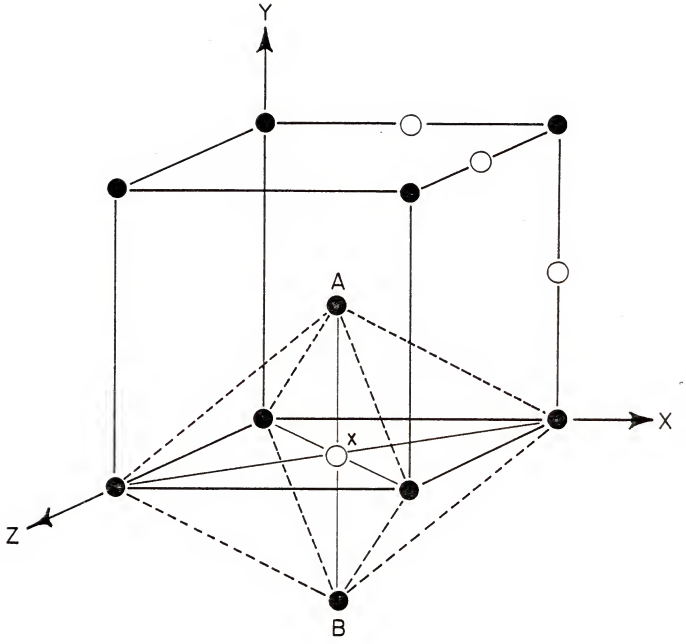
2.2 Mechanisms Responsible for the Appearance of the Yield Point Return

There are two primary mechanisms responsible for the observed yield point return in BCC metals: Snoek and Cottrell strain aging. In Cottrell strain aging, interstitial atoms diffuse to a dislocation and take up positions about it so as to reduce the strain fields associated with both the interstitial atoms and the dislocation. This lowers the total strain energy of the lattice. A greater stress is now required to initiate yielding, since movement of the dislocations requires that they be separated from their Cottrell atmospheres [9, 10]. In Cottrell strain aging, interstitial atoms diffuse to a dislocation from positions in the lattice that are as many as 10^3 atom jumps away [11]. This process is preceded in time by Snoek strain aging where interstitial atoms merely reorder in the stress fields of the dislocations by undergoing single atom jumps.

Snoek strain aging can be explained by referring to the Snoek effect as it occurs in the unit cell of a BCC lattice with interstitial atoms in solid solution. In Figure 2, the solvent atoms occupy positions at the cube corners and at the cube center. The interstitial atoms occupy octahedral sites at the cube edges and cube faces. The size of an interstitial

Figure 2.

Unit cell of a BCC metal. Solvent atoms (black dots) occupy cube corners and cube center. Interstitial solute atoms (white dots) occupy octahedral positions at cube edges and faces.

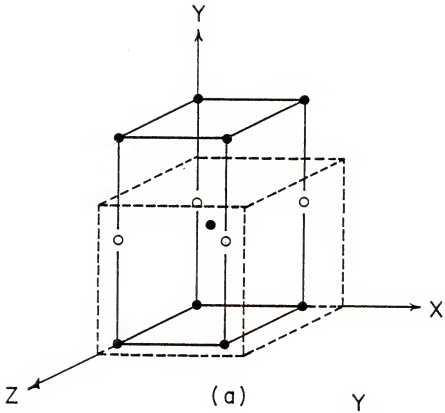

Y
A
X
x
Z
B

atom is larger than the space available for it in an octahedral site, such as position x. This causes substitutional atoms A and B to be displaced apart. If four of the octahedral positions lying parallel to the Y axis were to become occupied by interstitial atoms, as in Figure 3a, then the unit cell would become elongated in the Y direction and would assume a tetragonal shape. When no external stress is applied to a crystal lattice, a statistically equal number of interstitial atoms will occupy sites between atoms parallel to each of the X, Y, and Z axes, and the unit cell remains cubic. If one were to apply an external stress parallel to the Y axis, then the openings between atoms parallel to the Y axis would enlarge, and the opening between atoms perpendicular to the Y axis would decrease in size. It would then become energetically favorable for some atoms in position 1 in Figure 3b to shift to position 2. This is known as the Snoek effect or Snoek ordering, after Snoek who first postulated its existence in 1939 [12, 13].

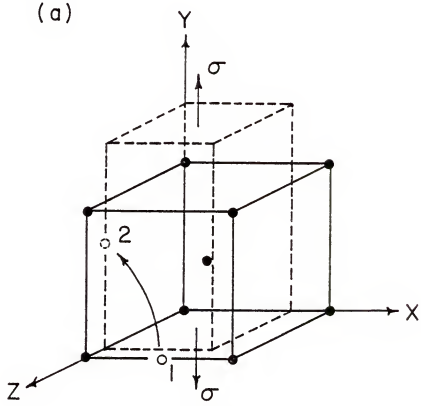
In the original treatment of the interaction between dislocations and interstitial atoms by Cottrell, an interstitial atom was assumed to act as a center of dilation in the lattice. Interstitial atoms were thus assumed to interact with edge dislocations only.

Figure 3.

Tetragonal distortion of unit cell.
(a) Four octahedral positions lying parallel to Y axis are occupied by interstitial atoms.
(b) External stress applied parallel to Y axis causes atom in position 1 to shift to more energetically favorable position 2.



(a)



(b)

Nabarro, in 1948 [14], and Cochardt, Schoek and Wiedersich, in 1955 [15], pointed out that, due to the tetragonal distortion around an interstitial atom, an interaction between the shear stresses around a screw dislocation with the interstitial atoms should also occur. Schoek and Seeger, in 1959 [16], then showed that it is possible to observe the Snoek effect in the strained region about a dislocation. Subsequently, Wilson and Russell, in 1959 [17], measured a return of the yield point due to Snoek ordering.

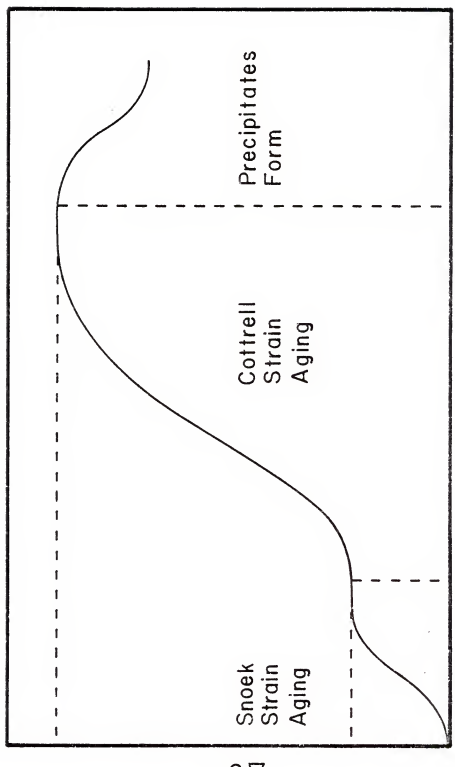
According to the 'breakaway' theory of yielding postulated by Cottrell and Bilby [9,10], yielding occurs when the stress level is sufficient to pull the dislocations from their interstitial atmospheres. On the other hand, the Johnston and Gilman theory of the yield point is based on the generation and multiplication of fresh dislocations, rather than on the breakaway of pinned dislocations [18,19]. Yielding, according to this latter theory, is observed when a sufficient number of dislocations move at a rate rapid enough for the plastic strain rate of the specimen to become equal to the imposed strain rate of the testing machine.

Evidence gathered to date indicates that it is the extent of aging that determines whether Cottrell-Bilby or Johnston-Gilman type yielding predominates. The extent of aging can be followed by monitoring the

increase of the yield point return, $\Delta\sigma$, with aging time, and a curve such as that shown in Figure 4 is typically obtained. It is seen that $\Delta\sigma$ (defined in Chapter IV) initially rises rapidly and then levels off to a nearly constant value. This first plateau is associated with the maximum amount of aging due to Snoek strain aging. After this initial plateau $\Delta\sigma$ again increases rapidly with time, and eventually levels off again. This latter region is associated with Cottrell strain aging. The second levelling off of the yield point return at long times is associated with saturation. Saturation occurs when the interstitial atoms have migrated to the dislocations in sufficient numbers to either relieve the strain energy induced in the lattice by the dislocations, or set up a concentration gradient opposing further migration to the dislocations [10]. Saturation may also occur when migration to the dislocations results in the depletion of interstitial atoms in the surrounding lattice. The concentration of interstitial atoms about the dislocations may be greater than the solid solubility limit of the solute in the solvent metal at that temperature, and precipitates may form. The decrease in yield point return, or softening, shown at very long times is believed to be associated with this precipitation process [20].

Figure 4

Schematic representation of the time dependence of the yield point return, $\Delta\sigma$, showing the Snoek and Cottrell strain aging regions.


Snoek
Strain
Aging
Cottrell
Strain
Aging
Precipitates
Form
Time
b
▽

The Cottrell-Bilby mechanism of the yield point is believed to operate when pinning is "weak", as in the Snoek strain aging region, or in the earliest stages of Cottrell strain aging; whereas, the Johnston-Gilman mechanism appears to operate at longer times, when the pinning is too strong for breakaway to occur [21,22,23].

The changes in the tensile stress-strain behavior of a BCC metal, as described in Figure 1, can also be related to the Snoek and Cottrell mechanisms. When aging occurs for times corresponding to the Snoek strain aging region, the flow stress level obtained upon deforming past the return of the yield point is the same as would have been obtained without an aging treatment. Upon aging at times corresponding to the early part of the Cottrell region, the flow stress level of the stress-strain curve is raised above that of a non-aged specimen. If a specimen is aged for times long enough for precipitation to occur, a decrease in the ductility of the specimen is observed [24].

2.3 The Unloading Yield Point

It is important to realize that strain aging is not the only process responsible for the reappearance of a yield point. When a specimen is deformed, unloaded, and then reloaded again, a yield point, referred to as an unloading yield point, may be observed [25]. These unloading yield points, most often observed in FCC metals [25,26,27,28], have also been observed in BCC metals [29].

Unloading yield points have been observed at temperatures too low for pinning of dislocations by solute atoms to occur. At these low temperatures, the magnitude of the unloading yield point has been found to be independent of temperature and to increase with increasing degree of unloading [26,27].

One theory advanced to explain the occurrence of the unloading yield point is based on dislocation-dislocation interactions. While the exact mechanism of this interaction has not been positively identified, it has been suggested that due to elastic interaction the dislocations rearrange themselves into low energy configurations upon unloading the specimen. A yield point occurs upon reloading, since plastic flow requires that energy be expended to move the dislocations out of these low energy configurations.

2.4 The Kinetics of Strain Aging

In section 2.2 it was shown that the Snoek effect is a process whereby application of a uniaxial tensile stress to a single crystal causes the interstitial atoms to redistribute themselves so that an excess number of solute atoms are found in sites between atoms aligned along a <100> direction most parallel to the applied stress. This redistribution does not

occur instantaneously, but develops with time. The rate at which the number of excess solute atoms arrive in the preferred sites is normally assumed to follow an exponential law [30],

$$\Delta n_p = \Delta n_p(\max)[1 - \exp(-t/\tau_\sigma)] \qquad (2.1)$$

where Δn_p is the number of excess solute atoms in the preferred sites at any time t, $\Delta n_p(\max)$ is the maximum number of excess atoms that may be redistributed into the preferred sites under a given tensile stress, and τ_σ is the relaxation time at constant stress.

If it is assumed that the magnitude of the yield point return is directly proportional to the number of atoms reordering in the stress field of the dislocations [17], then the time dependence of the yield point return for Snoek aging can be written

$$\Delta\sigma = \Delta\sigma(\max)[1 - \exp(-t/\tau_r)] \qquad (2.2)$$

where $\Delta\sigma$ is the magnitude of the yield point return obtained at any time t, and $\Delta\sigma(\max)$ is the maximum yield point return obtainable by the Snoek ordering mechanism.

The relaxation time, τ_r, for Snoek strain aging is the time required for the magnitude of the yield point return to reach 63.2% of its maximum value. The relaxation time, τ_r, has been substituted for τ_σ in the above expression since the two relaxation times are nearly equal for the case of interstitial atoms in BCC metals [30]. However, it is important to recognize that the relaxation time, τ_r, substituted for τ_σ, is the relaxation time that one obtains upon monitoring the Snoek effect by internal friction methods in a torsion pendulum. Substitution of this relaxation time, τ_r, into the above equation describing the time dependence of the yield point return is predicated on the assumption that the relaxation time during strain aging is the same as that observed during internal friction measurements.

The relaxation time, τ_r, measured by means of a torsion pendulum, can also be related to the diffusion coefficient of the solute atoms in the solvent metal by the equation [30]:

$$D = D_o[\exp(-Q/RT)] = a^2/36\tau_r \qquad (2.3)$$

where Q is the activation energy for diffusion and a is the lattice parameter of the solvent metal.

The time dependence of the yield point return in the Cottrell strain aging region was developed by Cottrell and Bilby [10]. In their treatment they showed

that the number of atoms arriving at a dislocation within time t is given by

$$n(t) = \alpha n_o (ADt/RT)^{2/3} \qquad (2.4)$$

where n(t) is the number of atoms that have segregated to a unit length of dislocation; n_o is the initial number of atoms in solution per unit volume; D is the diffusion coefficient for the interstitial atoms; α is a number nearly equal to 3; and A is a constant that depends on the elastic properties of the material, the degree of misfit of the solute atom, and the strength of the dislocation.

Assuming that the critical stress required to pull a dislocation from its atmosphere is proportional to the number of solute atoms in the atmosphere [31], then the magnitude of the yield point return should be proportional to $t^{2/3}$. This proportionality has been confirmed in numerous investigations for the early stages of aging by plotting the magnitude of the yield point return versus $t^{2/3}$ and obtaining a straight line.

The activation energy for the strain aging process is usually obtained in one of two ways. The first method assumes the Arrhenius relationship, or that the rate of the yield point return is proportional to exp(-Q/RT). Then, the data for the magnitude of the

yield point return are plotted either versus $t^{2/3}$ or versus t at each of several temperatures. The logarithm of the time required to obtain a specific value of the yield point return at a given temperature is then plotted versus the reciprocal of the absolute temperature. The activation energy for the process is then calculated from the slope of the resulting line.

A more rigorous method that is frequently used is that developed by Hartley [32], where the Cottrell-Bilby $t^{2/3}$ relationship was modified as follows:

$$\Delta\sigma/\bar{\sigma} = K_1 + K_2(Dt/T)^{2/3} \qquad (2.5)$$

where K_1 and K_2 are constants; $\Delta\sigma$ is the difference between the yield stress after aging and the prestrain flow stress; and $\bar{\sigma}$ is the arithmetic average of the yield stress after aging and the prestrain flow stress. This relation, like Cottrell-Bilby's, applies to the initial stage of the Cottrell strain aging region.

The activation energy for the strain aging process is determined by first plotting $\Delta\sigma/\bar{\sigma}$ versus $t^{2/3}$. The slope of each of these lines, S_1, is:

$$\text{slope } (S_1) = (K_2 D^{2/3})/T^{2/3} \qquad (2.6)$$

Upon taking the logarithm of both sides of this equation

one obtains:

$$\log (S_1 T^{2/3}) = \log K_2 + 2/3 \log D_o - 2/3 (Q/2.303\ RT) \quad (2.7)$$

Therefore, upon plotting $\log (S_1 T^{2/3})$ versus the reciprocal of the absolute temperature, one can calculate the activation energy, Q, of the strain aging process.

CHAPTER III
PREVIOUS INVESTIGATIONS

3.1 Summary

The earliest indications that the application of stress during aging could influence the strain aging response came from observations of the effect of applied stress on the initial yield point of iron. Subsequent studies revealed that the return of the yield point was also enhanced by the application of stress during aging. These studies were cursory in nature and were not able to disclose whether the observed enhancement was due to an increase in kinetics, resulting in the observation of a greater strain aging response in a given amount of time. These early investigations focused primarily on effects observed at times and temperatures corresponding to Cottrell aging. While some of these investigations included observations related to Snoek strain aging, the analysis of the strain aging kinetics is generally open to serious question. Similarly, some doubt is also cast upon the methods used to analyze data obtained from investigations of the effect of stress on the kinetics of the Cottrell strain aging.

In a number of strain aging investigations aging under stress was performed primarily as a matter of convenience. In these cases, aging was carried out by merely stopping the testing machine for a period of time and then continuing the deformation. This allows data to be gathered at aging times too short to be obtained by unloading the machine. Since it was not the intention of these investigators to study the effects of stress on strain aging, the information they generated relating to this matter is scanty. However, a number of these studies focused on Snoek strain aging. Several issues related to the nature of Snoek strain aging, in general, still need to be resolved. In particular, a significant discrepancy has been observed between experimental measurements of the temperature dependence and kinetics of the Snoek strain aging process, and that predicted theoretically.

In more comprehensive studies of the effects of stress on strain aging, the form of the relationship between aging stress and the strain aging response varies significantly. In some cases, the effect of raising the aging stress is to diminish the magnitude of the yield point return, rather than to increase it, and the conditions for obtaining a maximum value are not clear. The presence of an unloading yield point

and the large degree of stress relaxation that normally occurs during aging, when aging is performed under stress relaxation conditions, further complicate the analyses of the results that were obtained.

A consideration of these previous investigations with respect to each of these issues is thus important.

3.2 The Effects of Stress on the Kinetics of Strain Aging

The first indications that the stress required to initiate yielding could be increased by prior protracted application of stress came from observations made by Holden and Kunz [33] on iron single crystals. These crystals normally had either a very small or no initial yield point. However, when they were subjected to a tensile stress of 2/3 of the yield stress for one hour, a marked increase in the upper yield point was observed. They also observed that if these crystals were deformed and aged under tension at a stress just below the prestrain flow stress, a higher upper yield point was obtained than when aging occurred with the stress relaxed almost to zero.

Paxton [34] observed a similar increase in the initial yield stress of polycrystalline iron upon prestressing between the upper and lower yield stress, unloading, and reloading.

These initial studies focused almost exclusively on the initial yield point. Further evidence that strain aging could be enhanced by the application of stress during aging was obtained by Wilson and Russell [17]. They determined that aging a low carbon steel under stress resulted in a greater yield point return than aging in a stress-free condition. They also observed that while an increase in the concentration of interstitial carbon and nitrogen in solid solution increased the size of the yield point return, the yield point increased by a greater amount in those specimens that had been stressed during aging.

The first investigation of the effects of stress on the kinetics of strain aging was carried out by Brittain and Bronisz [35] on low carbon steel. Tests were performed at stress levels near the prestrain flow stress at times and temperatures corresponding to the initial portion of the Cottrell strain aging region. They found that the yield point return varied linearly with time rather than obeying the $t^{2/3}$ relationship predicted by Cottrell and Bilby [10]. Utilizing an Arrhenius relationship to describe the rate of strain aging, they obtained an activation energy, for specimens prestrained 4%, of 10,000 cal/mole. Similar analysis of data prestrained 8% and 12% resulted in activation energies of 9,400 and 8,500 cal/mole, respectively.

These values are about half those corresponding to the diffusion of carbon in iron. They interpreted these results as indicating an acceleration of the strain aging process.

This was challenged by Almond and Hull [36] who studied the effect of stress on strain aging in iron, niobium, and tantalum. An increase in the yield point return was observed when aging was carried out under stress over the entire Cottrell strain aging region. While aging was carried out at two stress levels for all three of the metals, only the data obtained for iron were analyzed for kinetics. The iron specimens were aged at stress levels corresponding to 8% and 71% of the flow stress obtained by prestraining 5%. The activation energies were obtained by using an Arrhenius relation where the logarithm of the time required to obtain a given increase in the upper yield stress was plotted versus the reciprocal of the absolute temperature. Four times and the corresponding values of the return of the upper yield point at the lower stress level, and, similarly, five pairs of values at the higher stress level were used. The activation energies obtained for the specimens aged at the lower stress level ranged from 17,400 ± 800 cal/mole to 18,800 ± 1600 cal/mole, while the activation energies for the

specimens aged at the higher stress level ranged from 19,100 ± 1400 cal/mole to 19,500 ± 1100 cal/mole. In addition, several tests were performed on specimens that had been allowed to relax in the grips of the testing machine without unloading. Similar analysis of the data obtained from these tests yielded an activation energy of 17,600 ± 1400 cal/mole. Almond and Hull therefore concluded that the diffusion of nitrogen controlled the strain aging process, and that the effect of stress was insignificant with respect to the kinetics of the process.

Several subsequent studies of the kinetics of strain aging were not concerned with the effects of stress on strain aging per se, but utilized aging under stress in order to be able to monitor strain aging at very short times where strain aging due to the Snoek ordering process could be observed. Rosinger, Craig, and Bratina [37] studied the kinetics in iron containing 0.01% between 273 K and 373 K. Aging under stress was performed by interrupting deformation for given intervals of time, and then continuing deformation to produce a yield point. The yield points thus obtained were plotted versus time to the 2/3 power. For each testing temperature, it was observed that the data appeared to define two stages: each stage being

linear with $t^{2/3}$, but having a different slope. Rosinger et al. then assumed that the first stage corresponded to Snoek strain aging, and the second to Cottrell strain aging. They further assumed that the time corresponding to the inflection between the two stages represented the time required for a carbon atom to undergo a single jump. This is because the Snoek strain aging process is essentially complete in that time. They checked this assumption by plotting the logarithm of the inflection time versus the reciprocal of the absolute temperature. This curve was then compared to a plot of the logarithm of the carbon atom jump time versus 1/T, and they concluded that the agreement was good. It is difficult to see, however, how a 24% difference between the slopes of the two lines they plotted can be considered to comprise good agreement. Actually, if one calculates the carbon atom jump time at 273 K on the basis of accepted diffusion data [38,39], a value of about 21 seconds is obtained, which does not agree well with the inflection time of about 65 seconds obtained by Rosinger et al.

Two methods were used by these investigators to analyze the kinetics of the first stage of strain aging. In the first, an activation energy was derived from the slope of the log inflection time verus 1/T plot, which was 15.5 kcal/mole. The second method consisted of applying Hartley's method [32] to plots of the yield

point return versus $t^{2/3}$. While a $t^{2/3}$ relationship can be predicted for the second stage of strain aging on the basis of the Cottrell-Bilby theory, the application of this theory to the first stage is highly questionable. The Snoek effect actually follows an exponential relationship with time. The activation energy obtained by this method was 14.5 kcal/mole. These two values for the activation energy of the first stage of strain aging are considerably below that for the diffusion of carbon in iron. Rosinger, Craig and Brattina concluded that the activation energy barrier for diffusion was lowered by the stress applied during aging.

Upon applying Hartley's method to the second stage of strain aging, an activation energy of 18.5 kcal/mole was obtained. This value is in good agreement with the activation energy of interstitial bulk diffusion in iron.

The analyses of two other investigations by Rosinger and Craig [40], in one case, and by Rosinger [41], in the other, are also subject to doubt. Both of these investigations followed the same experimental procedures used in the previous investigation, and in both cases strain aging was concluded to occur in two stages.

Rosinger and Craig also analyzed the kinetics of the first stage of strain aging by applying Hartley's

method to plots of the yield point return versus $t^{2/3}$. Unfortunately, their decision as to which of the data belonged to the first stage was based on the observed inflection times. For example, data were included in the analysis of the first stage which were obtained by strain aging at 333 K for times that would have permitted approximately not one but 200 atomic jumps to have occurred. The activation energy thus obtained was 6.6 kcal/mole, a value even lower than that obtained earlier.

The data of Rosinger and Craig for the second stage of strain aging could not be fitted to a $t^{2/3}$ relationship. Instead, they were fitted to a $t^{1/3}$ relationship and Hartley's method was applied, even though this method is based on the Cottrell-Bilby theory which predicts a $t^{2/3}$ relationship. This analysis yielded an activation energy of 17.6 kcal/mole.

Rosinger applied the same experimental techniques to both single crystal and polycrystalline specimens of iron. The methods of analysis previously described were also used. An activation energy of 14.1 - 14.9 kcal/mole was obtained for the first stage of strain aging for both the single crystal and polycrystalline specimens. An activation energy of 21.3 - 23.2 kcal/mole was obtained for the second stage of strain aging for the single crystal specimens, and a

value between 18.4 - 18.7 kcal/mole was measured for the polycrystalline specimens.

Duval and Dickson [42] performed a more detailed study of the influence of aging stress on the yield point return of mild steel. Specimens were aged under stress by unloading to a desired stress level and allowing stress relaxation to occur for aging times varying between 30 and 600 seconds. The aging stress was defined as the stress level at the beginning of the aging period. The kinetics of strain aging were obtained for specimens aged for 300 seconds between 282 K and 333 K, which correspond to a time in the Cottrell strain aging region at these temperatures. The specimens were aged at 0%, 80%, and 100% of the flow stress obtained at a 14% prestrain.

The yield drop was assumed to obey a kinetics equation of the type $\Delta\sigma/\sigma_f = A + Bt^n$, where A was assumed to represent an athermal component. Upon plotting the logarithm of the yield point return versus 1/T, L-shaped curves were obtained; the yield point return initially decreased linearly with decreasing temperature, then leveled off and remained constant with decreasing temperature. The constant value of the yield point obtained at the lower temperatures was taken as the value of A. This was then subtracted out and the slopes of the resulting lines were used to obtain the activation

energies. For specimens aged at 0%, 80%, and 100% of the flow stress, respectively, activation energies of 92 ± 20 kJ/mole (21.9 kcal/mole), 113 ± 12 kJ/mole (26.9 kcal/mole), and 88 ± 12 kJ/mole (20.9 kcal/mole) were obtained. They also applied Hartley's analysis to specimens aged at the flow stress. This yielded an activation energy of 84 ± 8 kJ/mole (20.1 kcal/mole). While the activation energies obtained for specimens aged at 0% and 100% of the flow stress are similar, and agree with values reported for the diffusion of carbon or nitrogen in iron, a higher activation energy was obtained for aging at 80% of the flow stress. It was also observed that the maximum yield point return obtained for aging at 300 K for 120 and 600 seconds occurred at 80% of the flow stress. Duval and Dickson concluded that this higher activation energy indicated that no single thermally activated process was rate controlling for aging performed near this stress.

Similar analyses were performed for specimens aged at both zero stress and at the flow stress for 30 seconds. The temperature range that was used was not specified; however, since some data are presented for specimens aged at 266 K and 300 K, it is probable that these were used in the analysis. A calculation of the jump times for carbon at these temperatures indicates that aging for 30 seconds at 266 K corresponds

to a time in the Snoek strain aging region, while aging for 30 seconds at 300 K corresponds to a time in the initial stages of the Cottrell strain aging region. It is possible, then, that the analysis of the kinetics for specimens aged 30 seconds utilized data from both the Snoek and Cottrell strain aging regions. The activation energies obtained for aging at zero stress and at the flow stress were, respectively, 54± 8 kJ/mole (12.9 kcal/mole) and 50 ± 8 kJ/mole (11.9 kcal/mole). No activation energy was obtained for any intermediate level of aging stress. These values are considerably lower than those corresponding to specimens aged at 300 seconds; however, Duval and Dickson claim they are in good agreement with those obtained by Rosinger [41], and Rosinger et al. [37], whose work has already been discussed. The validity of these values is questionable for the reasons already presented.

3.3 The Temperature Dependence of the Snoek Form of Strain Aging

The first investigation of the effect of temperature and time on the yield point return involving Snoek ordering of interstitial atoms in the stress fields of dislocations was made by Nakada and Keh [43]. They measured the return of the yield point in iron-nitrogen single crystals between 228 and 273 K using a

range of times sufficient to enable monitoring of both the initial and final stages of strain aging.

Data were presented showing the variation of the yield point return with time at five test temperatures. The magnitude of the yield point return was observed to initially increase with time and then level off to a nearly constant value. This plateau corresponds to the maximum amount of strain aging due to the Snoek mechanism, and should be independent of the testing temperature [16]. While, as expected, the time for the onset of the Snoek plateau decreased as the test temperature was raised, the magnitude of the yield point return in the Snoek plateau region changed with temperature. Upon raising the test temperature from 228 K to 236 K, the height of the Snoek plateau was slightly increased. A further increase to 248 K then decreased the height to a value approximately the same as that obtained at 228 K. The result of increasing the test temperature further was to decrease the Snoek plateau height significantly. The height of the Snoek plateau obtained at 273 K was about 47% lower than that obtained at 261 K and about 60% lower than that obtained at 248 K.

Similar observations were made by Delobelle, Oytana and Varchon [44] for the niobium-oxygen system.

They determined the variation of the magnitude of the yield point return with time at 344 K, 371 K and 398 K. For specimens tested at 371 K, the magnitude of the yield point return obtained in the Snoek plateau region was about 58% below that obtained for specimens tested at 344 K. At 398 K the height of the Snoek plateau region was essentially reduced to zero; that is, no strain aging due to the Snoek mechanism was observed, even though the process should have nearly reached completion at this stage. An extension of this work by Delobelle and Oytana [45] indicate a similar pattern for the tantalum-oxygen and iron-carbon-nitrogen systems. Data were obtained for the tantalum specimens at 298 K, 333 K, 363 K, and 393 K. At each successively higher temperature, the height of the Snoek plateau dropped below that obtained at the lower temperature. Thus, at 393 K the height of the Snoek plateau was 65% below that obtained at 298 K. A similar effect on raising the test temperature of the iron specimens was observed as with the niobium and tantalum specimens.

The above effects are not necessarily confined to the Snoek strain aging region. In several investigations the maximum yield point return obtained in the Cottrell strain aging region either increased slightly or remained about the same with increasing

temperature. However, in other investigations the maximum yield point return obtained in the Cottrell strain aging region decreased markedly with increasing temperature.

The results of Szkopiak and Miodownik [46], who strain aged commercially pure niobium between 373 K and 435 K in the Cottrell strain aging region show that increasing the test temperature increases the maximum yield point that is obtained. Similar studies made on a mild steel [47], and on a high strength low alloy steel [48], indicated that the maximum yield point return either increased slightly with temperature or remained about the same.

In contrast are results shown by Rosinger and Craig [40] for strain aging in the Cottrell region of a low carbon polycrystalline iron alloy. The effect of temperature on the time dependence of the yield point return was monitored and the results show that a maximum yield point return was obtained at 323 K and 333 K. From the shape of the curves obtained at lower temperatures, it appears that a smaller maximum yield point return was obtained at 323 K and 333 K than would be obtained at the lower temperatures if aging was carried out for longer times. The maximum yield point return obtained at 333 K was 24% lower than the magnitude of the yield point return obtained at 304 K upon aging for the same

amount of time. An increase in the test temperature should normally cause an increase in the value of the yield point return obtained after aging for the same interval of time, since at the higher temperature a greater number of atoms will arrive at a dislocation in a given interval of time.

Rosinger, Craig and Bratina [27] studied the strain aging of polycrystalline iron at higher temperatures. Their results indicate that a decrease in the maximum yield point return with increasing temperature was even more pronounced from 353 K to 373 K. At 373 K the maximum yield point return was 55% lower than that obtained at 323 K.

3.4 A Comparison between the Experimental and Theoretical Time Dependence of the Snoek Strain Aging Process

The time dependence of Snoek strain aging, as discussed in section 2.4, is normally assumed to follow the relationship

$$\Delta\sigma = \Delta\sigma_{max}[1 - \exp(-t/\tau_r)] \qquad (3.1)$$

where $\Delta\sigma_{max}$ is the maximum yield point return obtainable by Snoek ordering, and τ_r is the relaxation time of the Snoek ordering process. The relaxation time, τ_r, is the

time required for the yield point return to reach 63.2% of its maximum value, and the Snoek strain aging process should be essentially complete in the time it takes an atom to undergo a single jump.

This relationship was developed in accordance with the time dependence of the Snoek effect, as it occurs during internal friction measurements, where the driving force for the ordering process results from the tetragonal distortion in a crystal by the application of a very small external stress. It has generally been assumed that when the driving force for Snoek ordering results from the stress field of a dislocation, the magnitude of the yield point return will follow the same relationship with time. If this is true, the relaxation time determined from experimental measurements of the yield point return during Snoek strain aging should be the same as that obtained from torsion pendulum measurements of the Snoek effect. While results from several Snoek strain aging studies support this conclusion, there also exists significant evidence that contradicts it.

Wilson and Russell [17] investigated Snoek strain aging in a low carbon steel. Specimens were aged at 261 K under a reduced load between 80% and 90% of the prestrain flow stress. The rise in the yield point return was observed to be about 63% complete in 100

seconds, which is in agreement with the relaxation time for the ordering of carbon in iron at 261 K as measured by internal friction methods.

Nakada and Keh [43] investigated Snoek strain aging in iron-nitrogen single crystals in the temperature range of 228 K - 273 K. Aging was carried out at a stress level only sufficient to maintain good alignment of the specimen (5% of the yield stress). The results that were obtained indicate that aging was essentially complete in the time necessary for the nitrogen atoms to undergo a single atom jump.

In these studies it appears that the time dependence of the Snoek form of strain aging is the same as that of the Snoek effect as monitored by internal friction methods.

Delobelle, Oytana, and Varchon [44] studied Snoek strain aging in the niobium-oxygen system. Their experimentally measured relaxation times at 344 K, 371 K, and 398 K indicate that the kinetics of the Snoek ordering process during strain aging occur about 2.3 - 2.4 times faster than is predicted on the basis of the diffusion coefficients measured in a torsion pendulum.

Snoek strain aging in the tantalum-oxygen and iron-carbon-nitrogen systems were also studied by Delobelle and Oytana [45]. The dependence of the

yield point return on time in the tantalum-oxygen system was obtained at 298 K, 333 K, 363 K, and 393 K. A comparison of their experimental relaxation times with the corresponding torsion pendulum relaxation times indicates that the difference between the two relaxation times increases with decreasing testing temperature. At 393 K, the internal friction relaxation time is 2.7 times longer than the experimentally measured value. At 363 K, 333 K, and 298 K, the torsion pendulum relaxation times are, respectively, 3, 11.8, and 7.5 times longer than the experimentally measured relaxation times. The decrease in the difference between the experimental and calculated relaxation times observed with decreasing test temperature from 333 K to 298 K may have resulted from Delobelle and Oytana'a delineation of the Snoek plateau, since there is scatter in their data. Had the height of the Snoek plateau for their 333 K specimens been chosen at $\Delta\sigma = 1.65$ instead of 1.45, which is not an unreasonable choice, the corresponding relaxation time would be 80 seconds instead of 50 seconds, and the kinetics would appear to proceed only 7.4 times faster than the theory predicts, instead of 11.8 times faster.

Similarly, for the iron-carbon-nitrogen system, Delobelle and Oytana show a double Snoek plateau for specimens tested at 273 K, 253 K and 243 K. The

first plateau was assumed to result from the ordering of nitrogen atoms, and the second from the ordering of carbon atoms. The experimental relaxation times observed at 63.2% of the height of the Snoek plateau, associated with nitrogen at 273 K, 253 K, and 243 K, are respectively, 1.7, 3.9, and 4.6 times shorter than those calculated from diffusion coefficients. The difference between the experimental and calculated relaxation times for strain aging due to carbon, however, does not appear to change significantly with temperature. In fact, considering the degree of accuracy with which one can define the height of the Snoek plateau, the difference between the two relaxation times is probably insignificant.

3.5 The Stress Dependence of the Yield Point Return

The first investigations of the stress dependence of the yield point return focused on the initial yield point of iron as discussed in section 3.1. There, prestressing was performed either at a single stress level, or within a narrow range of stresses, and the results were then compared to aging under zero stress.

Hutchison and Louat [49] also measured the increase in the initial yield stress of iron after preloading below the yield point over a somewhat wider

range of stresses between 55.2 MPa and 110.4 MPa. They determined that the resulting increase in the yield stress varied linearly with the preload stress level and that there was a preload stress below which no significant increase in the yield point occurred.

Mura and Brittain [50] studied the effect of the application of stress during aging on the yield point return of ingot iron. They aged a set of specimens for a constant time of 2800 seconds, and observed the development of heterogeneous yielding as the aging stress was increased from 0 - 212 MPa. Their results indicated that the yield point became more fully developed; that is, showed more of the characteristics of both an upper and lower yield point as the aging stress was raised.

Mura, Lautenschlager, and Brittain [51] studied the effect of aging stress on iron specimens prestrained 4% at 303 K. Aging was carried out at four stress levels in the range of 20.7 MPa - 24.1 MPa, and the variation of the yield point return with time was monitored. The shapes of the curves that they obtained indicate that aging was carried out through all the stages of the Cottrell strain aging region, including saturation. Increasing the aging stress increased the magnitude of the yield point return over the time interval investigated.

Almond and Hull [36] investigated the effect of applying a tensile stress during the strain aging of iron, niobium, and tantalum. The upper yield stress, lower yield stress, yield point elongation, and flow stress level after yielding were monitored over all of the Cottrell strain aging region from the initial stages through saturation. Each metal was aged at two stress levels after prestraining 5%. The iron specimens were aged at 305 K at stress levels of 8% and 71% of the prestrain flow stress. Tantalum specimens were aged at 433 K at 4% and 71% of the prestrain flow stress, and the niobium specimens were aged at 403 K at 7% and 80% of the prestrain flow stress.

The maximum upper yield stress of the iron specimens aged at the higher stress level was 22% higher than that obtained upon aging at the lower stress level. Similarly, the maximum upper yield stresses of the niobium and tantalum specimens, aged at the higher stress level, were 37% and 14% higher, respectively, than the values obtained on aging at the lower stress level. The magnitude of the lower yield stress and the yield point elongation were also observed to increase more rapidly with time upon aging at the higher stress levels. The aging stress level was not observed to have any influence on the flow stress level obtained after yielding.

The most extensive study of the dependence of the yield point return on aging stress in BCC metals was performed by Duval and Dickson [42]. Mild steel was aged under stress by unloading to the desired stress level and allowing the specimen to relax for the desired aging time. The aging stress was taken as the stress level at the start of the aging period. However, the average stress level during aging, upon unloading to stresses above 70% of the prestrain flow stress, should be considerably lower than that at the beginning of the aging period. This is evident upon considering that Duval and Dickson defined the internal stress of the specimens during aging as that level at which the rate of stress relaxation was effectively zero, which was as low as 70% of the prestrain flow stress.

One set of specimens was prestrained 2%, 6%, and 14%, and aged at 266 K and 300 K for 30 seconds at several stress levels. Two relationships between the magnitude of the yield point return and the aging stress were found. For specimens aged at 300 K after prestraining 6%, the magnitude of the yield point return was observed to initially decrease with increasing aging stress to 40% of the prestrain stress. Upon aging at stress levels greater than 40%, the magnitude of the yield point return increased until it attained a maximum value at 60% of the prestrain flow stress. Aging at

stress levels above 60% of the prestrain flow stress caused the magnitude of the yield point return to then decrease until a minimum was obtained at 90% of the prestrain flow stress. A further increase in the aging stress caused the magnitude of the yield point return to increase to a value at 100% of the flow stress, about equal to that obtained at 60% of the prestrain flow stress. Only a minor difference was observed in the manner by which the yield point return varied with aging stress when the specimens were prestrained 2% and 14% instead of 6%.

On the other hand, for specimens aged at 266 K, raising the aging stress increased the magnitude of the yield point return up to a maximum value at an aging stress level approximately 70% of the prestrain flow stress. A further increase in aging stress up to 80% of the prestrain flow stress caused the yield point return to decrease and a value about equal to that obtained upon aging at 60% of the prestrain flow stress was found. Upon increasing the aging stress up to 100% of the prestrain flow stress, the magnitude of the yield point return continuously increased. At this point the yield point return was about equal to the maximum value obtained upon aging at 70% of the prestrain flow stress. In addition to aging for 30 seconds at each of these temperatures, Duval and Dickson

varied the aging time. Results were obtained for aging at 300 K for 10, 30, 120, and 600 seconds. Upon aging for 30 seconds, the magnitude of the yield point return decreased continuously with increasing aging stress to a minimum value at 100% of the prestrain flow stress. The effect of aging for 30 seconds yielded similar results, except for the appearance of a slight hump in the data at about 85% of the prestrain flow stress. The results obtained for this group of specimens do not compare with the results previously described for specimens also aged for 30 seconds at 300 K. This is probably due to a difference in the efficiency of their quench since batches were quenched by breaking the annealing tubes in water. The concentration of the solute in solid solution of steel is very sensitive to the efficiency of quenching and this should affect the degree of aging obtained.

Upon aging for longer times, the effect of raising the aging stress was to increase the magnitude of the yield point return up to a maximum value observed at 85% of the prestrain flow stress. The effect of increasing the aging time from 120 to 600 seconds was to increase the height of the observed peak.

These results were interpreted by Duval and Dickson as demonstrating not only the occurrence of strain aging, but also the presence of an unloading

yield point and a degree of relaxation during aging. At the lower temperatures, where an increase in aging stress causes the magnitude of the yield point return to decrease, the unloading yield point was suggested to be the primary influence; whereas at higher temperatures and longer times, strain aging was assumed to be the dominant influence. Upon aging at the higher temperature for a shorter period of time, a combination of both effects was observed. In addition, Duval and Dickson claimed that the maximum yield point return was obtained at a stress level they called the internal stress, where the rate of relaxation was observed to be zero, since, at that level, a minimum amount of dislocation motion would favor pinning of the dislocations by the solute atoms.

While the focus of this dissertation is strain aging in BCC metals, and all the investigations reviewed so far have been concerned with strain aging in BCC metals, some of the strain aging investigations of HCP metals are of interest with respect to the results obtained when aging was performed under stress.

In HCP metals the distortion around an interstitial atom in an octahedral site is not sufficiently asymetric to induce Snoek ordering in the stress fields of the dislocations. Solute atom pairs are then commonly invoked to explain the strain aging effects observed in these metals.

Malik and Dickson [52] investigated strain aging in the titanium-oxygen system. Specimens were aged at 363 K, 405 K, and 449 K, at stress levels between 0% and 100% of the flow stress obtained upon prestraining 8%. The effect of raising the aging stress level was to decrease the magnitude of the yield point return down to a minimum at approximately 50% of the prestrain flow stress. A further increase in the aging stress caused the magnitude of the yield point return to then increase, with the degree of increase being greater at higher temperatures.

In addition, a number of specimens were aged at 496 K. An increase in the aging stress level was accompanied by an increase in the magnitude of the yield point return for all values of aging stress. A maximum yield point return was obtained at 100% of the prestrain flow stress. The greatest increase in the yield point was observed between 20% and 80% of the prestrain flow stress; that is, S-shaped curves were obtained for plots of the magnitude of the yield point return versus aging stress.

These results were interpreted by Malik and Dickson as indicating that the unloading yield point was the predominant mechanism operating when the specimens were unloaded to zero stress levels. The results obtained for specimens aged at low temperatures

were explained on the basis of dislocation rearrangements. They pointed out that due to the large degree of relaxation which occurred during aging at the higher stress levels, the dislocations were able to rearrange themselves into low energy configurations and give rise to a yield point upon reloading. The results obtained upon aging at higher temperatures were interpreted as being indicative of strain aging, and at intermediate temperatures, both strain aging and dislocation rearrangements were assumed to play a role.

A similar study of a zirconium-oxygen alloy was performed by Dickson and Sayar [53]. The experimental method used was similar to that used by Duval and Dickson, and by Malik and Dickson, where a large degree of relaxation was observed during aging under stress. Upon aging at 423 K - 493 K, an increase in the aging stress level was accompanied by a decreasing magnitude of the yield point return down to a minimum at 60% of the prestrain flow stress. Further increases in the aging stress level were accompanied by an increasing yield point return. For specimens aged at 573 K, an increase in the aging stress level was accompanied by an increase in the magnitude of the yield point return up to a maximum at 80% of the prestrain flow stress. Further increases in the aging stress level were accompanied by a sharp decrease in the magnitude of

the yield point return. Upon aging at 623 K, the magnitude of the yield point return was much lower than that obtained at 573 K at almost all aging stress levels. The effect of increasing the aging stress level was to continuously increase the magnitude of the yield point return.

These results were all obtained upon aging for 2 minutes. After aging for 10 minutes, similar results were obtained with the exception that the aging stress level corresponding to the minimum yield point return shifted to lower values as the temperature was increased. Also, for data obtained upon aging at 623 K, there appeared as many maxima and minima in the plot of the yield point return versus aging stress, as there were data points.

The mechanisms suggested to explain the results were similar to those given by Malik and Dickson, in that the unloading yield point and dislocation rearrangements during relaxation predominate at low temperatures, and that strain aging predominates at high temperatures.

Finally, the importance of the effects of recovery on the strain aging process was illustrated in a study by Kelly and Smith [54] on a zirconium-oxygen alloy. Specimens were prestrained and unloaded to a value approximately half of the ultimate tensile

strength. The variation of the yield point return with temperature indicated that increasing the temperature from 523 K to 623 K decreased the strain aging response. These results were interpreted by Kelly and Smith as being indicative of the process of recovery, since TEM observation revealed a decrease in the dislocation density at the higher temperature.

CHAPTER IV
EXPERIMENTAL METHOD

4.1 Specimens

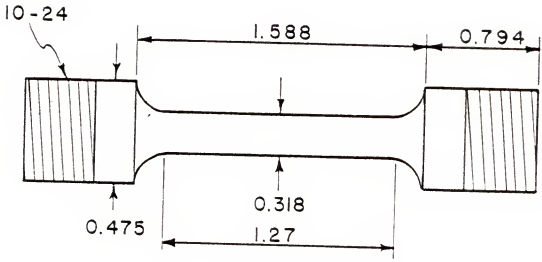
Vanadium rods of 12.7 mm. diameter, 99.9% pure, and tantalum rods of 6.35 mm. diameter, 99.98% pure, were obtained from the Materials Research Corporation. The rods were swaged to a diameter of 4.75 mm. and cylindrical specimens were machined having dimensions shown in Figure 5a. The specimens were pickled in a bath of 50:50 nitric acid and hydrofluoric acid to remove grease due to machining and handling, and an oxide layer that might diffuse into the specimens during recrystallization. The specimens were annealed in groups of eight in a Precision Scientific MINIVAC vacuum annealing furnace under a dynamic vacuum of 10^{-4} torr. The vanadium specimens were annealed for one hour at 1373 K to give a uniform grain size of 58 μm. The tantalum specimens were annealed for one hour at 1473 K to yield a uniform grain size of 79 μm.

Initial plans called for all the testing to be done with vanadium. As testing proceeded, and the vanadium stock neared depletion, an attempt was made to obtain another lot of vanadium. A heat was obtained from the Materials Research Corporation that gave a smaller

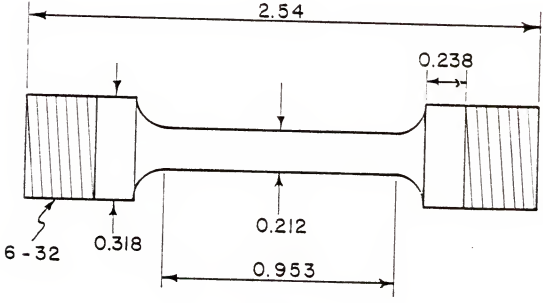
Figure 5.

Specimen dimensions used in this investigation.

All dimensions in cm.



(a)



(b)

grain size on recrystallization than the previous heat under the same annealing conditions, and an initial yield stress that was lower than that of the previous heat. Although analysis revealed that the heat contained more oxygen than the previous heat, no yield point return was obtained even upon aging at higher temperatures for long times. Analysis indicated that the heat contained 7500 ppm titanium. Shikama, Ishino and Mishima [55] have determined that titanium acts as a scavenger of oxygen in vanadium. From their data it was determined that 7500 ppm titanium is sufficient to remove all the oxygen present from solution.

A small amount of the original heat in the form of 6.35 mm. rods was then obtained. These had previously been swaged from 12.7 mm. rods and charged with various amounts of hydrogen. These rods were swaged to 3.18 mm. and cylindrical tensile specimens were machined having dimensions shown in Figure 5b. The specimens were annealed at 1373 K to give a uniform grain size of 54 μm. An additional soak of 4 hours at 1023 K was needed to remove the hydrogen that had been charged into the metal. After this treatment, the initial yield stress and the work hardening rate during deformation matched that of the original uncharged heat and the hydrogen was considered removed.

4.2 Composition

The analyses supplied by the manufacturer for the two lots of materials that were used are given in Table I. These analyses are batch analyses which the manufacturer claimed were typical of the lots, and are not analyses of the lots themselves. It was therefore necessary to obtain an independent analysis for the interstitial content of the material used. Since these metals take up oxygen readily at high temperatures, this analysis was obtained after recrystallization. The interstitial contents are given in Table II.

The effect of hydrogen on the measured yield point return can be considered negligible since the dynamic strain aging region of hydrogen in both vanadium and tantalum occurs at a temperature range that is 150 K - 200 K below the testing temperatures used [56]. The randomizing effect of thermal vibrations at the test temperatures is therefore assumed sufficient to counteract any ordering or drift tendency by the hydrogen atoms in response to the presence of dislocations.

While the carbon content of the vanadium that was used was almost as great as the oxygen content, and the activation energies for the diffusion of these interstitials in vanadium are similar [57], the solubility of carbon in vanadium at the test temperatures

TABLE I

Materials Research Corporation Batch Analysis of Vanadium and Tantalum

	Vanadium	Tantalum
C	15	20
O	220	65
H	6	9
N	35	30
Al	10	<10
Mg	<10	<10
Ca	<10	10
Ti	<10	<10
Fe	100	25
Cu	<10	<10
Si	400	15
Cr	<30	<30
Ni	10	<10
Mo	<30	<30
Sn	<30	<30
Sb	<30	--
Pb	<30	<30
V	rest	.24
Zr	--	10
Nb	--	<30
Ag	--	10

TABLE II

Analysis of Interstitial Content of Vanadium and Tantalum

	Composition after Recrystallization*	
	Vanadium	Tantalum
Oxygen	132	52
Carbon	124	10
Nitrogen	383**	12

* Analyzed by Gollob Analytical Service

** Lot analyzed by Materials Research Corporation before recrystallization

employed is believed to be extremely low [58]. Boratto [59], using internal friction methods to measure interstitial diffusivities in vanadium, was not able to obtain a Snoek peak due to carbon, even though a sufficient amount of carbon was present in the material. Since Snoek peak height measurements are indicative of the amount of solute present in solid solution, the carbon was assumed to be tied up as vanadium carbides. The carbon content of the vanadium used in Boratto's study was approximately 60 ppm. It is therefore assumed that the carbon content in the vanadium used in this investigation, 124 ppm, did not contribute significantly to the magnitude of the yield point return.

The nitrogen content in the vanadium used in this investigation can be assumed to be sufficient to give a sizeable yield point return upon strain aging. However, the activation energy for the diffusion of nitrogen in vanadium is considerably greater than that of oxygen. A comparison between the jump times of a nitrogen and an oxygen atom, at the test temperatures which were used, is made in Table III. Since the longest aging time used in this study was ten hours, it is apparent that nitrogen did not contribute significantly to the magnitude of the yield point return obtained. Strain aging of vanadium, in this investigation, can therefore be assumed to be due solely to the presence of oxygen.

TABLE III

Jump Times of Oxygen and Nitrogen in Vanadium and Tantalum

Vanadium*		
Temperature (K)	Oxygen	Nitrogen
353	68.6 min.	28 weeks
363	21.3 min.	7 weeks
373	7.0 min.	2 weeks

Tantalum*		
Temperature (K)	Oxygen	Nitrogen
368	20.49 sec.	452 weeks
378	7.89 sec.	115 weeks
388	3.20 sec.	31 weeks

*Jump times were calculated using diffusion coefficients determined by Boratto [59].

A similar comparison between the jump times of nitrogen and oxygen in tantalum can be made. Since the longest aging time used during the strain aging study of tantalum in this investigation was 120 minutes, then, on the basis of the information provided in Table III, the influence of nitrogen can be assumed to have been negligible. The activation energy for the diffusion of carbon in tantalum is approximately the same as that for nitrogen [60]. Strain aging of the tantalum used in this study can therefore be assumed to be solely due to oxygen.

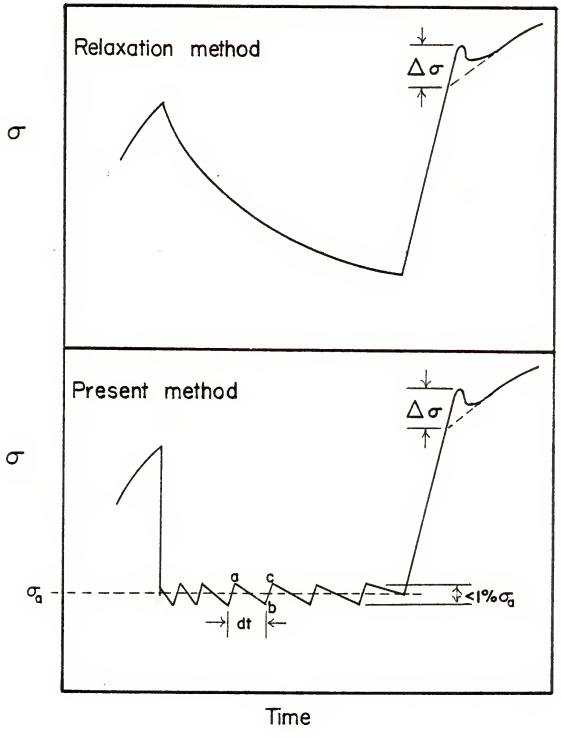
4.3 Equipment

Tensile tests were performed on a floor model C/D INSTRON Universal Testing Instrument. The specimens were maintained at the testing temperature during prestraining, aging, and restraining, by using an inverted tensile fixture suspended in an agitated silicone oil bath. During the test period the oil bath was maintained to within ± 1 K at each test temperature.

The method used to hold the stress (load) constant during aging differs significantly from that used by other investigators. Figure 6 illustrates the primary difference. In the technique used by others in the past, referred to here as the relaxation method, the specimen is loaded to a desired prestrain and the Instron

Figure 6.

Comparison between the relaxation method and the present method of applying the aging stress. In the relaxation method the stress falls continuously with time. In the present method the stress can be held to within <0.5% of the aging stress.


Relaxation method
Δσ
σ
Present method
Δσ
σ
σa
a
c
b
dt
≤1%σa
Time

crosshead is stopped so that the specimen is permitted to relax during aging. Alternatively, the specimen may be unloaded from the flow stress to a desired aging stress (load) and then allowed to relax. The primary shortcoming of this method is that the stress is not maintained at a constant level but falls progressively with time. The range of stress levels the specimen relaxes through depends on the material used, the testing temperature, prestrain strain rate, and the stress level at the beginning of relaxation. Stress relaxation ranges as large as 50% - 60% of the flow stress level at the beginning of relaxation have been reported in strain aging studies [53].

This problem has been largely eliminated in the present investigation by utilizing an electronically controlled feedback loop between the Instron strip chart and crosshead. In this method, a specimen is prestrained the desired amount and unloaded to a preset aging load. When the preset aging load is reached the crosshead automatically begins reloading the specimen until a preset value is reached which is only about 2 lbs. (8.90 N) higher than the load which caused the crosshead to begin reloading. The crosshead movement then stops until the specimen has relaxed down to the preset aging load and the cycle is repeated. It was thus possible to

maintain the stress (load) level constant to within <0.5% of the prestrain flow stress, or to within 1% of the aging stress.

Another advantage of the method used was that it was possible to measure both the amount of strain and the strain rate during aging. This may be understood by referring to Figure 6 which shows the shape of the load-time relationship during aging. In a cycle such as defined by points a, b, and c, the specimen deforms plastically between points a and b, while the load relaxes. The machine then reloads the specimen rapidly between points b and c. During the reloading interval between points b and c, the strain can be considered elastic. With the aid of measurements of the magnitude of this load increment, and with the aid of the combined spring constant of the machine and specimen, a calculation of this elastic increment of strain can be made.

The relationship between the elastic strain and plastic strain portions of the cycle can be developed in the following manner. The specimen and the load cell are viewed as a spring and specimen connected in series as shown in Figure 7a. When the crosshead motion is stopped, the total extension of the spring and specimen is maintained constant. Relaxation of the specimen by plastic deformation results in a shortening of the spring in order to maintain the constant extension, as shown in

Figure 7.

Schematic representation of specimen and testing machine connected in series.
(a) Crosshead stops and total extension remains constant.
(b) Specimen relaxes and spring shortens to maintain total extension.

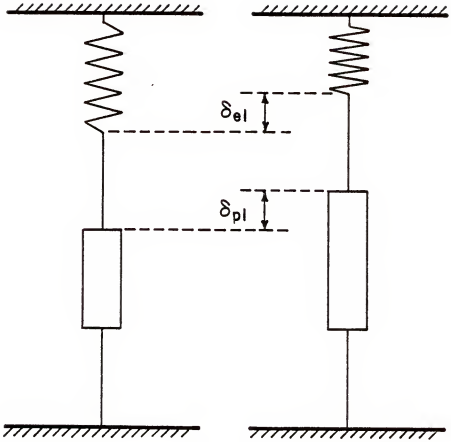

δ_{el}
δ_{pl}
(a)
(b)

Figure 7b. This shortening of the spring is displayed by the load cell as a drop in the load. Since,

$$\delta_{plastic} = \delta_{elastic} \tag{4.1}$$

where $\delta_{plastic} = L(\varepsilon_{pl})$ and $\delta_{elastic} = \Delta P/K$, then

$$L\ (\varepsilon_{pl}) = \Delta P/K \tag{4.2}$$

and therefore,

$$\varepsilon_{pl} = \Delta P/KL \tag{4.3}$$

The total strain for the complete aging period is then taken equal to the strain per load drop times the number of load drops. The strain rate at any time during the aging can be evaluated by dividing a strain increment by the time increment required to relax the load.

4.4 Test Method

The testing procedure used for both the vanadium and tantalum specimens was similar, and can be understood by referring to Figure 8.

A specimen was deformed past the initial yield point and luders band into the region of homogeneous strain to a value of prestrain, ε_{pre},

Figure 8.

Schematic representation of stress (load)-strain (time) diagram defining the parameters σ_f, σ_a, ε_{pre}, t, and $\Delta\sigma$.

Stress
Load
σ_f
σ_a
ϵ_{pre}
t
$\Delta\sigma$
σ_f'
Strain
Time

defined as the difference between the initial upper yield point and that point on the stress-strain curve, σ_f, where the Instron crosshead was stopped prior to unloading. While raw load-time data were recorded autographically during the test, these were converted to stress-strain data by utilizing average gage section diameter and gage section length measurements that had been made on an optical comparator prior to testing. After prestraining the desired amount, the specimen was then unloaded to the desired aging stress, σ_a, which is normally expressed in this investigation as some fraction of the flow stress σ_f, obtained at the end of the prestraining; that is, the aging stress is commonly expressed as σ_a/σ_f. Upon unloading the specimen to the aging stress, the specimen was then aged for a period of time, t, which began when the aging stress was reached, and ended when reloading was commenced. At the end of the aging period the specimen was reloaded to obtain the yield point return.

Two other test variables were the testing temperature, which was kept constant throughout the entire test period, and the prestrain strain rate, which was the same used during reloading.

The return of the yield point was measured by the extrapolation method shown in Figure 8. In this method, the yield point return, $\Delta\sigma$, is defined as the difference between the upper yield point and the point,

defined as the difference between the initial upper yield point and that point on the stress-strain curve, σ_f, where the Instron crosshead was stopped prior to unloading. While raw load-time data were recorded autographically during the test, these were converted to stress-strain data by utilizing average gage section diameter and gage section length measurements that had been made on an optical comparator prior to testing. After prestraining the desired amount, the specimen was then unloaded to the desired aging stress, σ_a, which is normally expressed in this investigation as some fraction of the flow stress σ_f, obtained at the end of the prestraining; that is, the aging stress is commonly expressed as σ_a/σ_f. Upon unloading the specimen to the aging stress, the specimen was then aged for a period of time, t, which began when the aging stress was reached, and ended when reloading was commenced. At the end of the aging period the specimen was reloaded to obtain the yield point return.

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The return of the yield point was measured by the extrapolation method shown in Figure 8. In this method, the yield point return, $\Delta\sigma$, is defined as the difference between the upper yield point and the point,

σ_f', at which the flow curve obtained after yielding extrapolates back to the elastic portion of the stress-strain curve.

The extrapolation method was used since it effectively eliminates from consideration the work hardening that occurs during aging under stress. This has been confirmed by estimating the amount of work hardening during aging in terms of the strain developed during the aging period, and the average work hardening rate based on the work hardening rate before and after aging.

CHAPTER V
RESULTS

5.1 The Effects of Stress on the Kinetics of Strain Aging

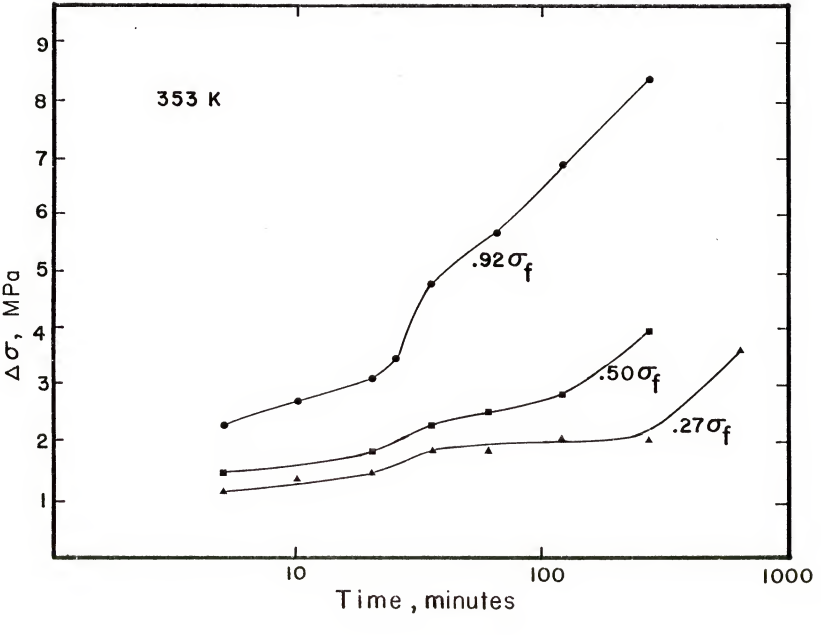
5.1.1 The Effect of Aging Stress on the Kinetics of Snoek Strain Aging in Vanadium

Figure 9 shows the effect of aging time on the magnitude of the yield point return for specimens prestrained 9% at a strain rate of $6.7 \times 10^{-5} s^{-1}$ and 353 K. Three curves are shown corresponding to specimens aged at 27%, 50%, and 92% of the prestrain flow stress.

Each curve can be considered to consist of 3 portions with respect to the time of aging. Initially, the magnitude of the yield point return increases with time, and then levels off in a plateau which is associated with the maximum yield point return obtainable by the Snoek strain aging process. The increase in yield point return observed at later aging times is associated with the Cottrell form of strain aging. It is evident that, at all aging times, the effect of raising the level of the aging stress is to increase the magnitude of the yield point return obtained.

Figure 9.

Effect of stress on the time dependence of the yield point return for vanadium aged at 353 K.


353 K
.92σf
.50σf
.27σf
Δσ, MPa
Time, minutes
1
2
3
4
5
6
7
8
9
10
100
1000

A portion of the observed yield points can be assumed to arise from the effect of unloading described in section 2.3. An assessment of the magnitude of this effect was made at room temperature where strain aging effects are not expected to influence the yield point obtained after reloading. A yield point return of 1.03 MPa was obtained upon unloading to $0.27\sigma_f$ and immediately reloading. Similarly, an unloading yield point return of 0.65 MPa was obtained for the $0.50\sigma_f$ level. Upon unloading to $0.92\sigma_f$ and reloading, no yield point was obtained.

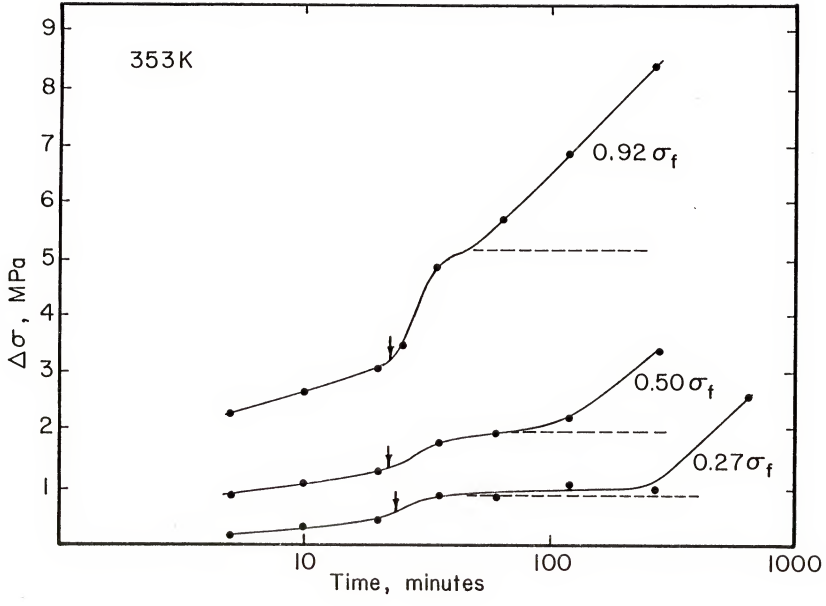
The data of Figure 9, corrected for this unloading point contribution, are shown in Figure 10. The vertical arrows correspond to the relaxation time of the Snoek strain aging process at each stress level, where the relaxation time is that required for the yield point return to reach 63.2% of the maximum value indicated by the dashed line. Figure 10 demonstrates that the time needed to reach the relaxation time does not change as the aging stress level increases.

5.1.2 The Effects of Stress on the Kinetics of Cottrell Strain Aging in Tantalum

Tantalum specimens were prestrained 9% at 378 K using a strain rate of $6.7 \times 10^{-5} s^{-1}$ and then

Figure 10.

Effect of stress on the time dependence of the yield point return for vanadium aged at 353 K. Data are corrected for effect of unloading yield point. Arrows correspond to experimental relaxation time.


353K
0.92 σf
0.50 σf
0.27 σf
Δσ, MPa
Time, minutes
1
2
3
4
5
6
7
8
9
10
100
1000

aged for 25 minutes at a number of stress levels between $20\%\sigma_f$ and $95\%\sigma_f$. The variation of the yield point return with aging stress is shown in Figure 11. Here it is observed that the effect of raising the aging stress level is to increase the magnitude of the yield point return continuously up to a maximum at $85\%\sigma_f$. Upon aging at stress levels above $85\%\sigma_f$, the magnitude of the yield point return decreases slightly. The yield point return obtained by aging at $85\%\sigma_f$ is approximately 4 times greater than that obtained upon aging at $20\%\sigma_f$.

Two methods were used to determine if the aging stress level had any effect on the activation energy of Cottrell strain aging. These were Hartley's and the Arrhenius method, described in section 2.4. Specimens were prestrained 9% at a strain rate of $6.7 \times 10^{-5} s^{-1}$ at 368 K, 378 K, and 388 K and aged at 20%, 50%, and $85\%\sigma_f$ between 5 and 120 minutes.

The results are plotted according to Hartley's method in Figures 12-15. In Figures 12, 13, and 14, representing data obtained at 20%, 50%, and $85\%\sigma_f$, respectively, the yield point increases linearly with $t^{2/3}$ at each of the test temperatures. The activation energies for strain aging, at each stress level, were determined as follows: First, the slopes of the lines, S_1, were obtained by applying linear regression analysis to the data in Figures 12, 13, and 14. The logarithm

Figure 11.

Stress dependence of yield point return for tantalum aged at 378 K for 25 minutes.

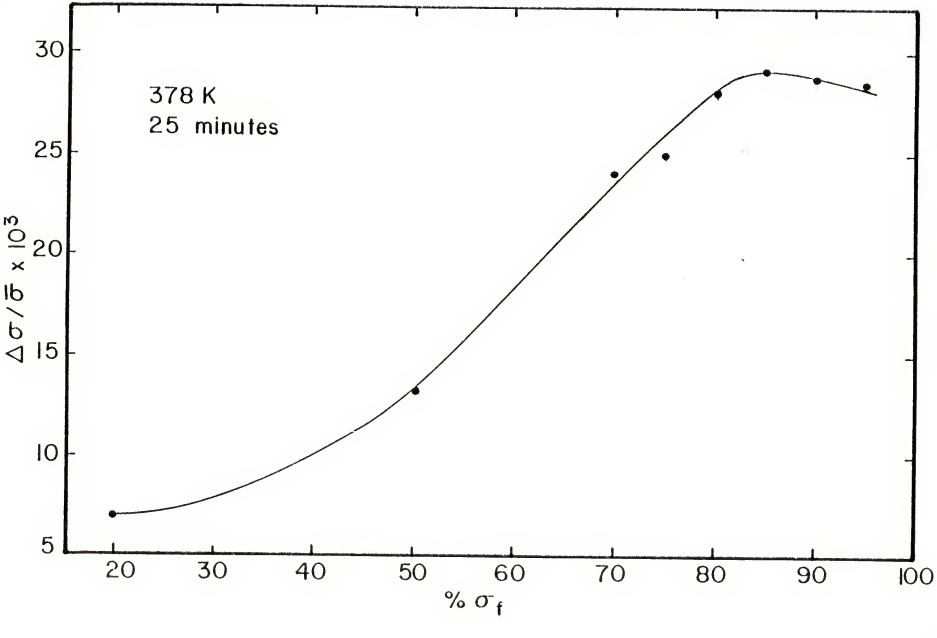

378 K
25 minutes
$\Delta\sigma/\bar{\sigma} \times 10^3$
30
25
20
15
10
5
20
30
40
50
60
70
80
90
100
% σ_f

Figure 12.

Yield point return versus $t^{2/3}$ for tantalum aged at $20\%\sigma_f$.

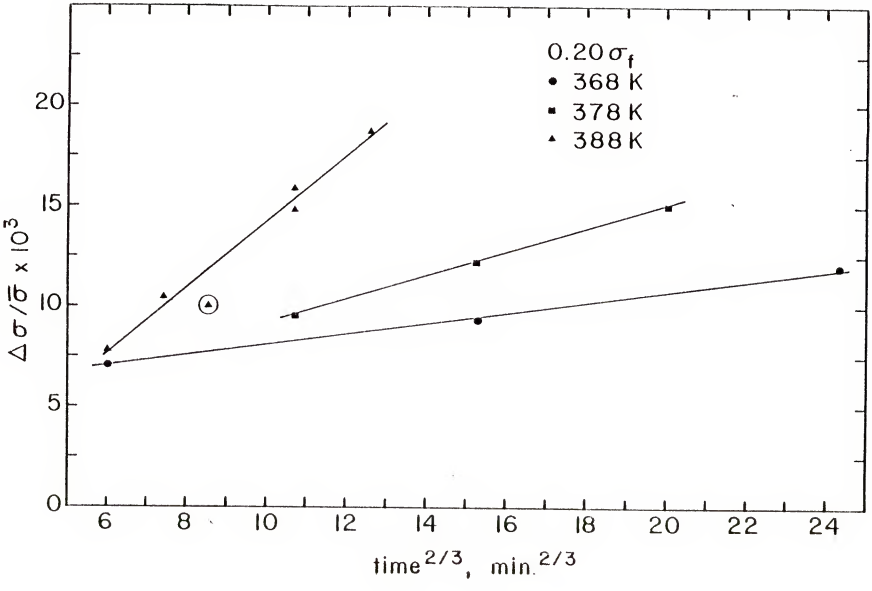

0.20 σ_f
368 K
378 K
388 K
$\Delta\sigma/\bar{\sigma} \times 10^3$
0
5
10
15
20
6
8
10
12
14
16
18
20
22
24
time$^{2/3}$, min.$^{2/3}$

Figure 13.

Yield point return versus $t^{2/3}$ for tantalum aged at $50\%\sigma_f$.

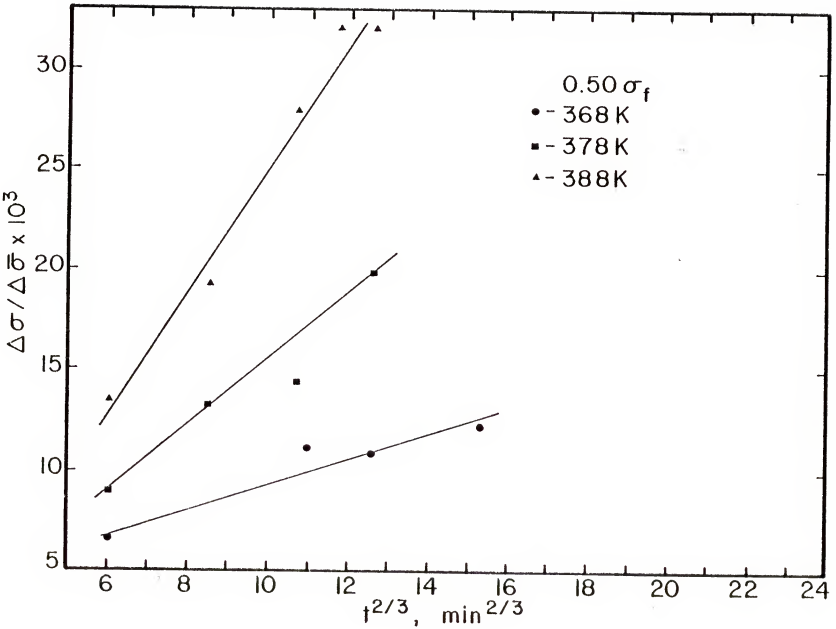
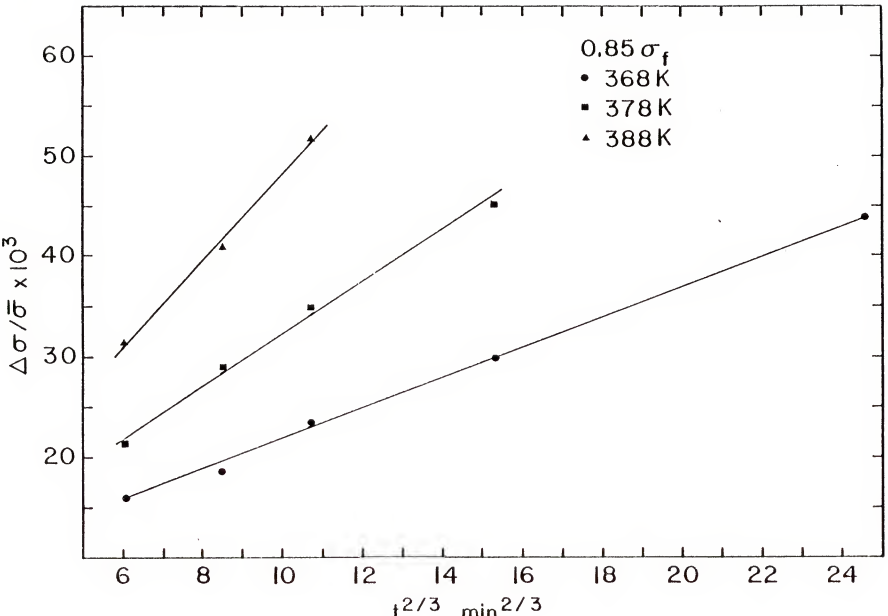

0.50 σ_f
● - 368 K
■ - 378 K
▲ - 388K
$\Delta\sigma/\Delta\bar{\sigma} \times 10^3$
$t^{2/3}$, $min^{2/3}$
5
10
15
20
25
30
6
8
10
12
14
16
18
20
22
24

Figure 14.

Yield point return versus $t^{2/3}$ for tantalum aged at $85\%\sigma_f$.


0.85 σ_f
• 368K
▪ 378K
▲ 388K
$\Delta\sigma/\bar{\sigma} \times 10^3$
60
50
40
30
20
6
8
10
12
14
16
18
20
22
24
$t^{2/3}$, $min^{2/3}$

of the product $S_1 T_a^{2/3}$, where T_a is the test temperature, was then plotted versus $1/T_a$ as shown in Figure 15. Thus, each line in Figure 15 represents results from each of the three aging stress levels. The activation energies calculated from these slopes appear in Table IV.

The results based on measurements of the time to attain a fixed value of $\Delta\sigma/\overline{\sigma}$ at various temperatures are shown in Figures 16-21. In Figure 16 it is seen that data for aging at $20\%\sigma_f$ indicate that the yield point return follows an approximately linear relationship with time at all three test temperatures. The logarithms of the times for the yield point return to attain the same value are plotted versus the reciprocal of the absolute temperature in Figure 17. The activation energy for strain aging was calculated from the slope of the resulting line and is tabulated in Table IV. The activation energies for strain aging of specimens aged at 50% and $85\%\sigma_f$ were obtained similarly using the data in Figures 18-21, and are also tabulated in Table IV.

5.2 The Temperature Dependence of the Snoek Form of Strain Aging in Vanadium

The temperature dependence of the Snoek form of strain aging is shown in Figure 22. In this diagram

Figure 15.

Determination of activation energy for tantalum aged at three stress levels.

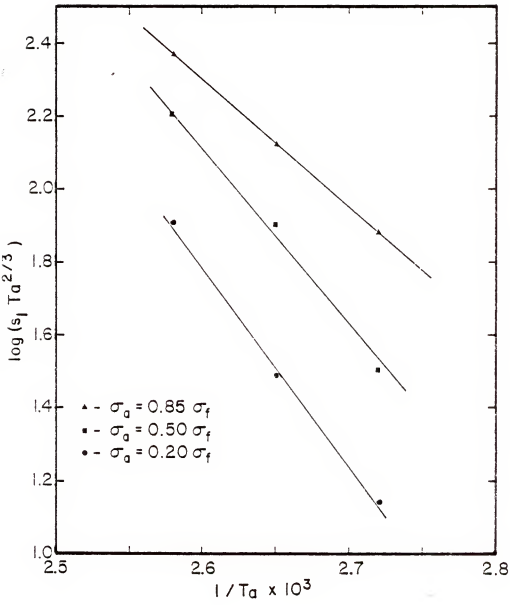

2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
2.5
2.6
2.7
2.8
log (s_l Ta^2/3)
1 / Ta x 10^3
▲ - σ_a = 0.85 σ_f
■ - σ_a = 0.50 σ_f
● - σ_a = 0.20 σ_f

Figure 16.

Yield point return versus time for tantalum aged at 20%σ_f.

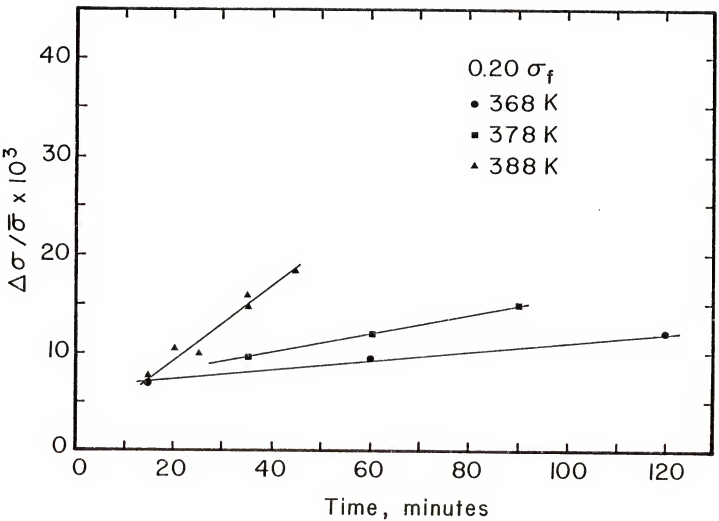

0.20 σ_f
• 368 K
▪ 378 K
▲ 388 K
$\Delta\sigma/\bar{\sigma} \times 10^3$
0
10
20
30
40
0
20
40
60
80
100
120
Time, minutes

Figure 17.

Logarithm of 1/t versus the reciprocal of the absolute temperature for tantalum aged at 20%σ_f.

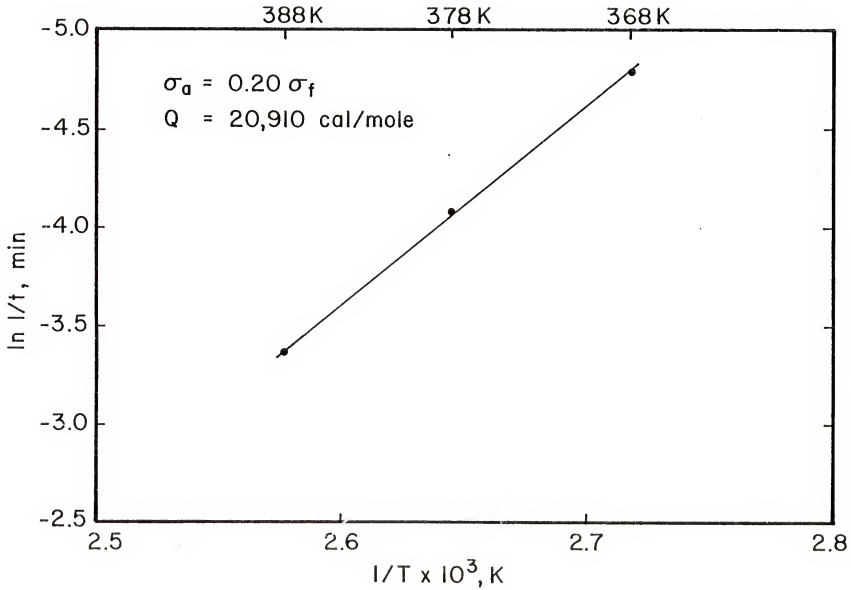

388K
378K
368K
-5.0
-4.5
-4.0
-3.5
-3.0
-2.5
$\sigma_a = 0.20\ \sigma_f$
Q = 20,910 cal/mole
ln 1/t, min
2.5
2.6
2.7
2.8
1/T x 10^3, K

Figure 18.

Yield point return versus time for tantalum aged at 50%σ_f.

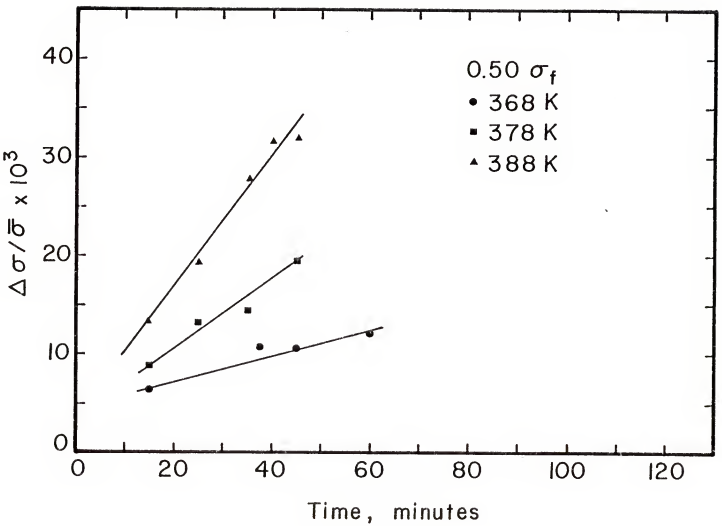

0.50 σ_f
• 368 K
▪ 378 K
▲ 388 K
$\Delta\sigma/\bar{\sigma} \times 10^3$
0
10
20
30
40
0
20
40
60
80
100
120
Time, minutes

Figure 19.

Logarithm of 1/t versus the reciprocal of the absolute temperature for tantalum aged at 50%σ_f.

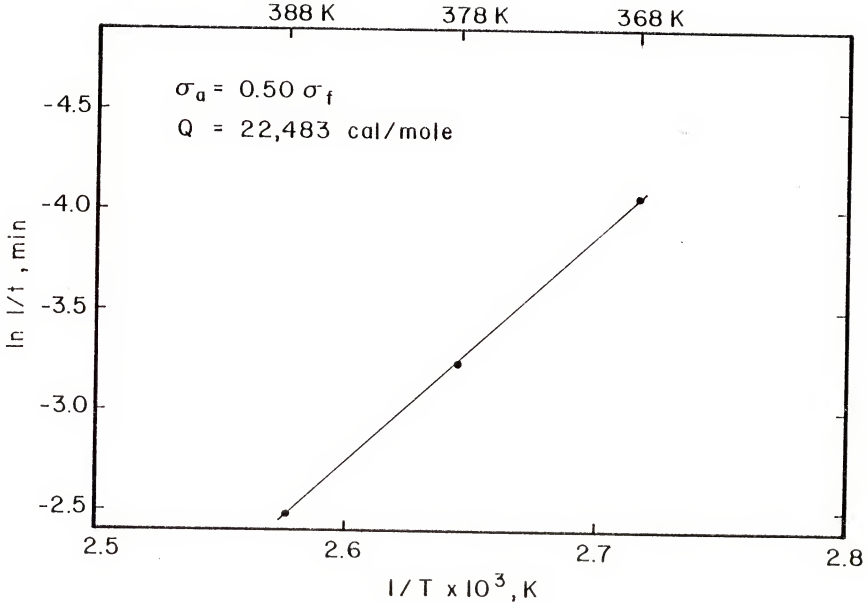

388 K
378 K
368 K
$\sigma_a = 0.50\ \sigma_f$
Q = 22,483 cal/mole
-4.5
-4.0
-3.5
-3.0
-2.5
ln 1/t, min
2.5
2.6
2.7
2.8
1/T x 10^3, K

Figure 20.

Yield point return versus time for tantalum aged at 85%σ_f.

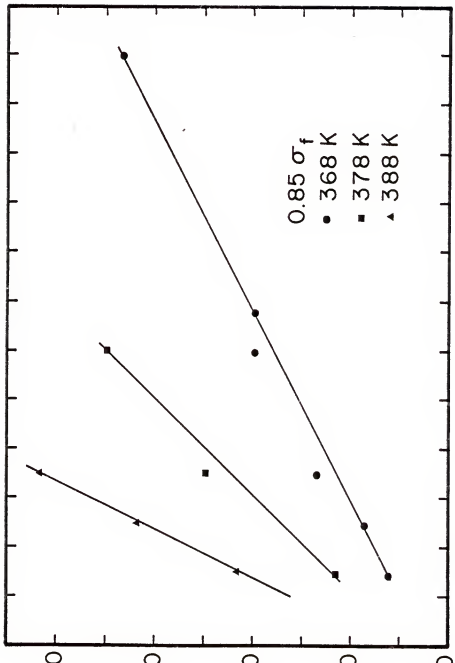

0.85 σ_f
368 K
378 K
388 K
$\Delta\sigma/\sigma_i \times 10^3$
Time, minutes
10
20
30
40
50
0
20
40
60
80
100
120

Figure 21.

Logarithm of 1/t versus the reciprocal of the absolute temperature for tantalum aged at 85%σ_f.

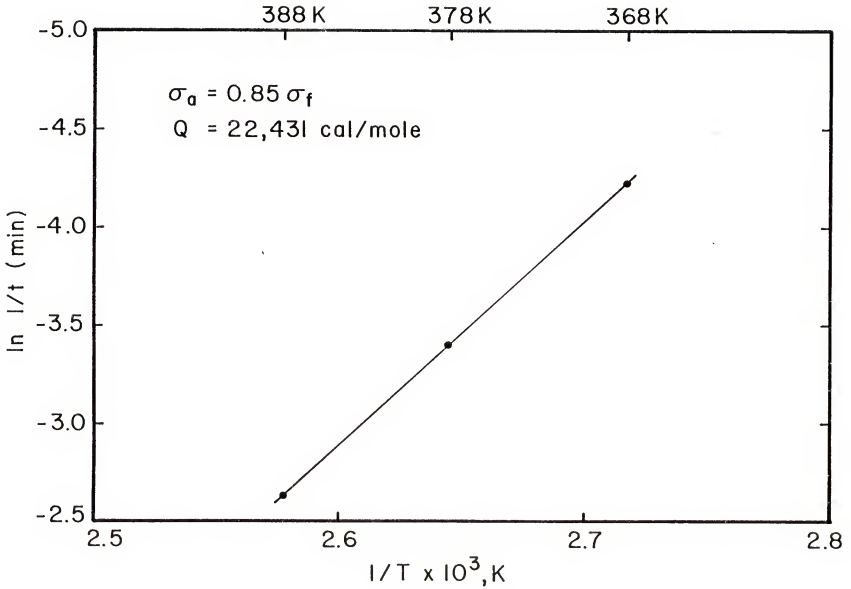

388 K
378 K
368 K
$\sigma_a = 0.85\,\sigma_f$
Q = 22,431 cal/mole
ln 1/t (min)
-5.0
-4.5
-4.0
-3.5
-3.0
-2.5
2.5
2.6
2.7
2.8
1/T x 10^3, K

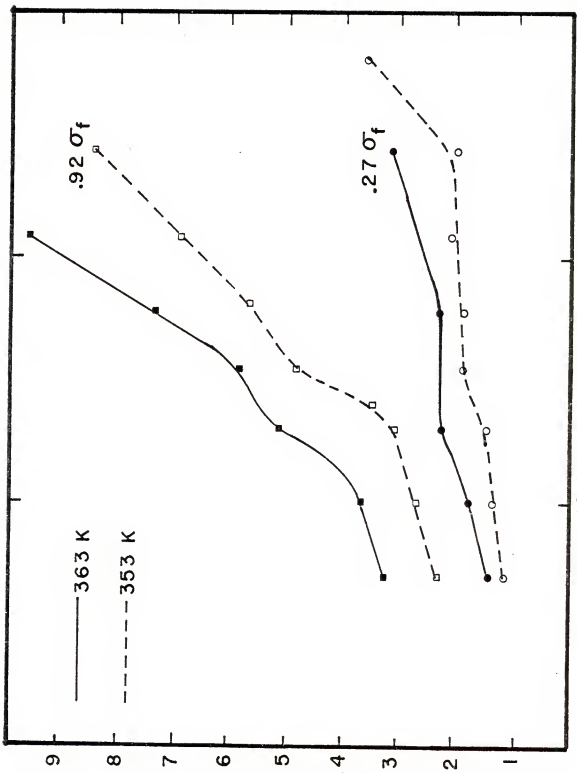
TABLE IV

The Effect of Aging Stress on the Activation Energy of Cottrell Strain Aging in Tantalum

Aging Stress, σ_a	Hartley's Method	Arrhenius Method
	Q (cal. mole)	
0.85 σ_f	23.320	22,431
0.50 σ_f	35,280	22,483
0.20 σ_f	38,443	20,910

Figure 22.

Effect of temperature on the time dependence of the yield point return for vanadium aged at $27\%\sigma_f$ and $92\%\sigma_f$.


363 K
353 K
.92 σ_f
.27 σ_f
$\Delta\sigma$, MPa
Time, minutes
1
2
3
4
5
6
7
8
9
10
100
1000

the variation of the yield point return with time is shown for specimens prestrained 9% at a strain rate of $6.7 \times 10^{-5} s^{-1}$ that were tested at two temperatures, 353 K and 363 K, and aged at two stress levels: 27% and 92%, of the prestrain flow stress.

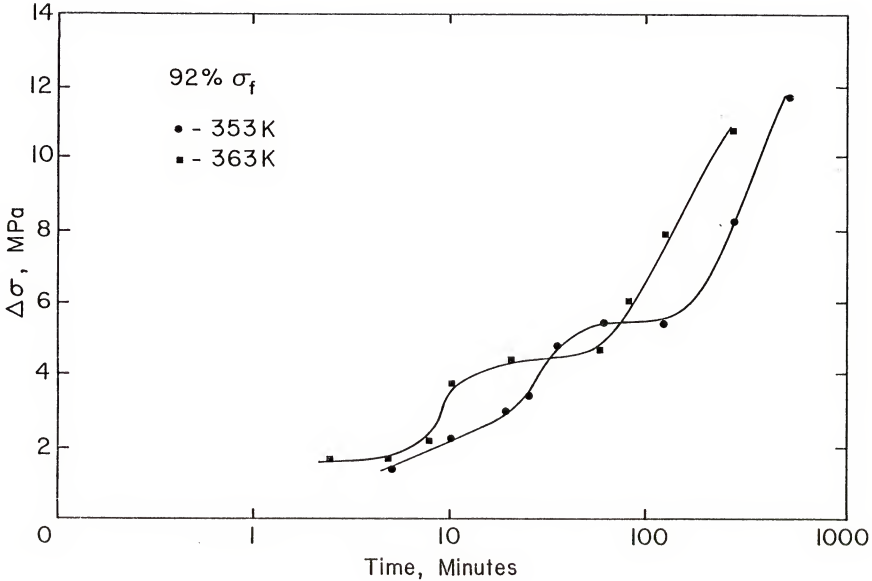
This figure reveals that raising the test temperature increases the magnitude of the yield point return over the entire range of aging time. The maximum extent of strain aging due to the Snoek mechanism at 363 K, as indicated by the height of the plateau region, is approximately the same, or only slightly higher than, that obtained for specimens tested at 353 K. This is true for both stress levels.

It is also observed that a shorter aging period is needed at 363 K to obtain the same magnitude of yield point return as obtained at 353 K. For specimens tested at 363 K, as at 353 K, an increase in the aging stress level has no effect on the kinetics of the Snoek strain aging process, as indicated by the time required for the onset of the plateau region at both stress levels.

Figure 23 shows the effect of test temperature on the variation of the yield point return with time for specimens that were annealed at a temperature and time too low for complete recrystallization to occur. The specimens were tested at 353 K and 363 K and aged at

Figure 23.

Effect of temperature on the time dependence of the yield point return for vanadium aged at 92%σ_f. Specimens were not fully recrystallized prior to testing.

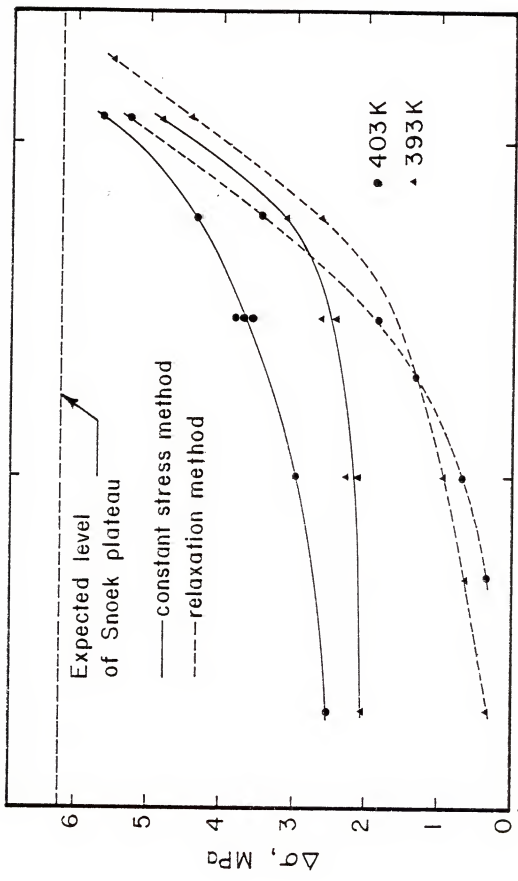

92% σ_f
• - 353K
▪ - 363K
Δσ, MPa
Time, Minutes
0
2
4
6
8
10
12
14
1
10
100
1000

92% of the prestrain flow stress. In contrast to the results obtained for fully recrystallized specimens, raising the test temperature does not increase the magnitude of the yield point return obtained at all aging times. For example, the height of the Snoek plateau region at 363 K is lower than that obtained at 353 K.

Figure 24 shows the results of an experiment performed to test a hypothesis, discussed in section 6.2, that Snoek dynamic strain aging was responsible for observations made by other investigators where the Snoek plateau height varied as 1/T. In this figure the variation of the yield point return with time is shown for specimens prestrained various amounts at a strain rate of $8.9 \times 10^{-5} s^{-1}$ and tested at 393 K and 403 K. The aging times utilized at these temperatures correspond to the end of the Snoek plateau and the beginning of the Cottrell aging region. At the time these tests were made only a few specimens of vanadium were available and it was necessary to perform more than one test on each specimen. One group of specimens was aged at a constant stress of $0.90\sigma_f$. Another group was permitted to relax during aging by merely stopping the crosshead in order to assess how much contribution to the diminution in the yield point is made using this method. In addition, specimens were

Figure 24.

Effect of temperature on the time dependence of the yield point return for vanadium. Solid lines indicate specimens aged under a constant stress of 90%σ_f. Dashed lines indicate specimens aged under relaxation.


Δσ, MPa
0
1
2
3
4
5
6
1
10
100
Time, seconds
Expected level
of Snoek plateau
constant stress method
relaxation method
403 K
393 K

aged for the same interval of time after different pre-strains to determine the effect of performing several tests on one specimen.

The solid curves in Figure 24 represent data obtained by aging at a constant stress level of $0.90\sigma_f$ and the dashed curves represent data obtained by simply stopping the crosshead and allowing the specimen to relax during aging. The data points shown at aging times of 10 and 35 minutes for specimens tested at $0.90\sigma_f$ represent various amounts of prestrain. The expected level of the Snoek plateau was estimated by assuming, on the basis of data shown in Figure 9, that Snoek strain aging at 353 K was complete between 45 and 60 minutes. Several specimens were then aged at a stress level of $0.90\sigma_f$ at 353 K for times between 35 and 90 minutes after prestraining 9% at a rate of $8.9 \times 10^{-5} s^{-1}$. The height of the Snoek plateau was then taken to be approximately 6.2 MPa, as indicated in Figure 24 by a dashed straight line.

For specimens aged at the constant stress level of $0.90\sigma_f$, it is observed that the height of the Snoek plateau at these temperatures is less than half that predicted on the basis of data obtained at the lower temperature. The height of the Snoek plateau obtained upon using the relaxation method is even lower, and the magnitude of the yield point return approaches zero at

short aging times. The effect of varying the amount of prestrain is seen to have little effect on the magnitude of the yield point return at these temperatures and times.

5.3 A Comparison Between the Experimental and Theoretical Time Dependence of the Snoek Strain Aging Process

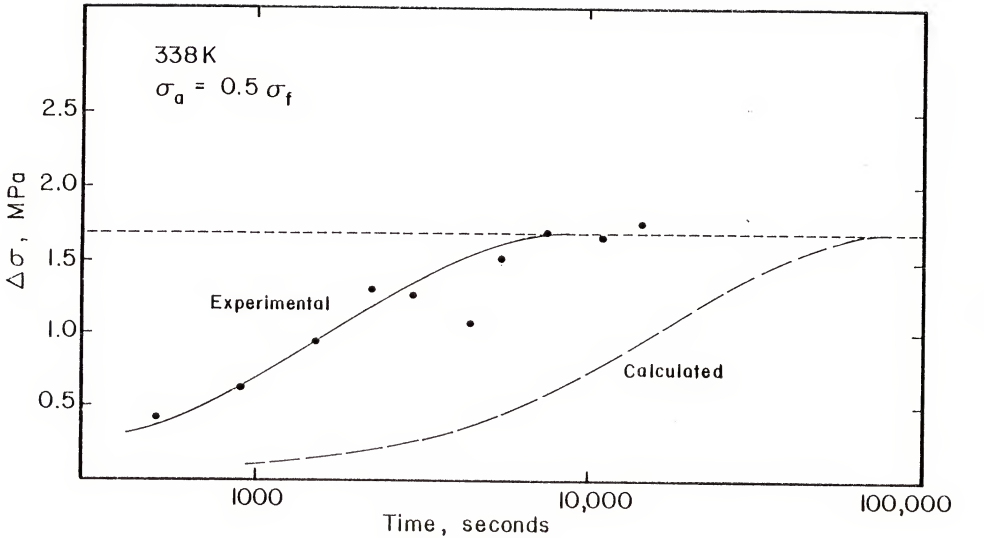
The experimental results of Figure 25 (solid line) show the time dependence of the yield point return for specimens aged at 338 K and 50% of the prestrain flow stress for 35 minutes. These data have been corrected for the effect of the unloading yield point; that is, the magnitude of the yield point obtained at room temperature upon unloading to $50\%\sigma_f$ and immediately reloading has been subtracted from the yield point measured after strain aging.

The theoretically predicted time dependence of the yield point return (dashed line) was calculated by using equation 2.2: $\Delta\sigma = \Delta\sigma(\max)[1 - \exp(-t/\tau_r)]$, with $\Delta\sigma_{max} = 1.69$ MPa, the maximum yield point return obtained experimentally, and a value of τ_r, 18,094 seconds, based on the diffusion coefficient of oxygen in vanadium.

The experimentally measured relaxation time corresponding to Snoek ordering at this temperature was found to be 1,850 seconds where τ_r was taken as the time for the yield point return to reach 63.2% of its maximum

Figure 25.

Comparison between the experimental and theoretical Snoek aging kinetics for vanadium aged at 50%σ_f at 338 K.


338K
$\sigma_a = 0.5\,\sigma_f$
2.5
2.0
1.5
1.0
0.5
$\Delta\sigma$, MPa
Experimental
Calculated
1000
10,000
100,000
Time, seconds

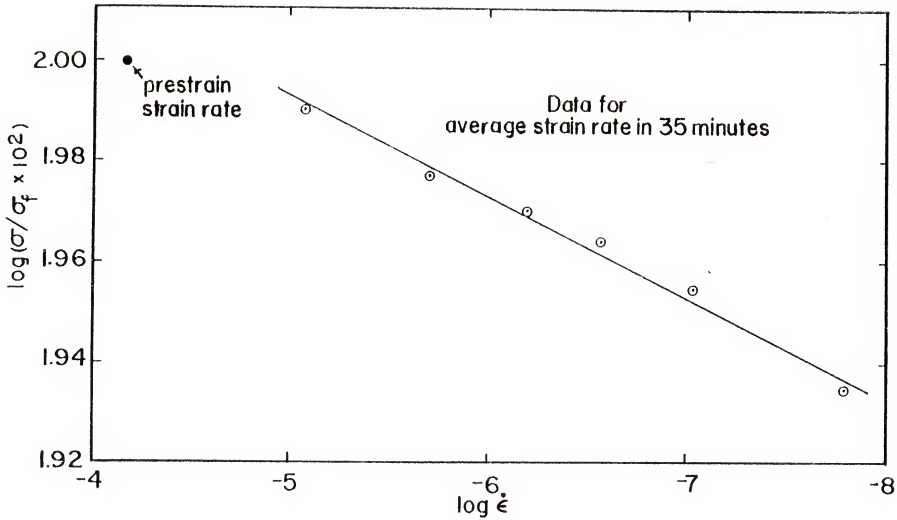
value. Comparison of this value with the value of τ_r used in the above calculation shows that, at this temperature, the Snoek effect occurs approximately 10 times faster during strain aging than predicted. A similar analysis of the data obtained by aging at 353 K and 363 K indicated that the relaxation times corresponding to Snoek aging were approximately half those predicted on the basis of internal friction diffusion coefficients.

5.4 The Effect of Temperature, Aging Time, Prestrain, and Prestrain Strain Rate on the Stress Dependence of the Snoek Form of Strain Aging in Vanadium

It was observed in this investigation that the specimens deformed at a measurable strain rate during aging at higher stress levels. The strain rate during aging was a maximum when aging occurred at stress levels approaching the prestrain flow stress, and decreased as the level of the aging stress decreased. Figure 26 shows that for aging stress levels between $0.86\sigma_f$ and $0.98\sigma_f$ the average strain rate over a 35 minute aging period at 353 K follows a power law with respect to aging stress. Thus, for example, the strain rate during aging at $0.92\sigma_f$ is found to be only about 2 1/2 orders of magnitude slower than the prestrain strain rate. As the aging

Figure 26.

Logarithm of the aging stress versus logarithm of the average strain rate over a 35 minute aging period for vanadium aged at 353 K.

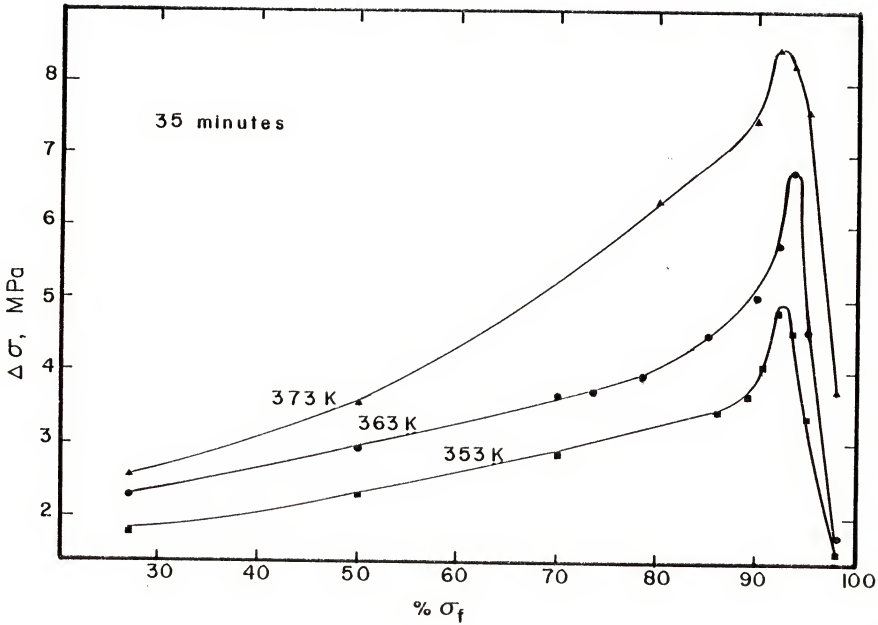

2.00
1.98
1.96
1.94
1.92
$\log(\sigma/\sigma_f \times 10^2)$
prestrain
strain rate
Data for
average strain rate in 35 minutes
-4
-5
-6
-7
-8
$\log \dot{\epsilon}$

rate approaches the prestrain flow stress, the strain rate approaches the prestrain strain rate.

Figure 27 shows the variation of the yield point return with aging stress, with the aging stress expressed as a percentage of the prestrain flow stress. The amount of prestrain, prestrain strain rate, and time of aging, were held constant, while the testing temperature and aging stress was varied. The specimens were prestrained to 9% at a rate of $6.7 \times 10^{-5} s^{-1}$ and aged for 35 minutes at 353 K, 363 K, and 373 K. Inspection of the data reveals that raising the level of the aging stress from 27% to 98% of the prestrain flow stress causes the yield point return to increase continuously up to a maximum value at 92% of the prestrain flow stress. At this aging stress level, the magnitude of the yield point return is approximately 3 times that obtained at the lowest stress level (27%). Aging at stress levels above 92% of the prestrain flow stress results in the magnitude of the yield point return dropping off precipitously. At 98% of the flow stress it approaches or falls below the value obtained upon aging at 27% of the flow stress. The level of aging stress corresponding to the maximum yield point return did not appear to shift significantly as the test temperature was varied between 353 and 373 K. It can also be

Figure 27.

Effect of temperature on the stress dependence of the yield point return for vanadium aged 35 minutes.


35 minutes
373 K
363 K
353 K
Δσ, MPa
% σf
8
7
6
5
4
3
2
30
40
50
60
70
80
90
100

observed that the effect of raising the test temperature is to increase the magnitude of the yield point return at all aging stresses.

A comparison of the strain rates measured during aging at the higher stress levels is shown in Figure 28 for all three temperatures. The data are seen to fall on a single line, indicating that the strain rate corresponding to a particular aging stress level is the same for all three temperatures.

Figure 29 shows the variation of the yield point return with aging stress for specimens prestrained 9% at a rate of $6.7 \times 10^{-5} s^{-1}$ and aged at 363 K. The amount of prestrain, prestrain strain rate, and temperature were held constant, while the period of aging time was varied. Results are given for specimens aged 35 minutes, corresponding to a time in the Snoek strain aging region at this temperature; and for 120 minutes, which corresponds to a time in the Cottrell strain aging region. This figure shows that at both 120 minutes and 35 minutes a maximum yield point return occurs for aging at 92% of the prestrain flow stress. The magnitude of the maximum at 120 minutes is about 4 1/2 times greater than at $27\%\sigma_f$. The yield point return again is seen to drop off precipitously at aging stress levels above $92\%\sigma_f$.

Figure 28.

Logarithm of the aging stress versus the logarithm of the average strain rate over a 35 minute aging period for vanadium aged at three temperatures.

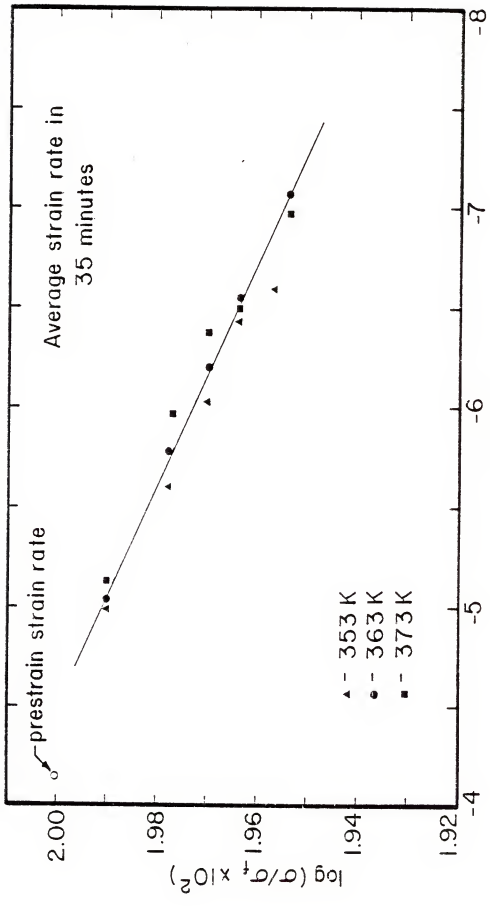
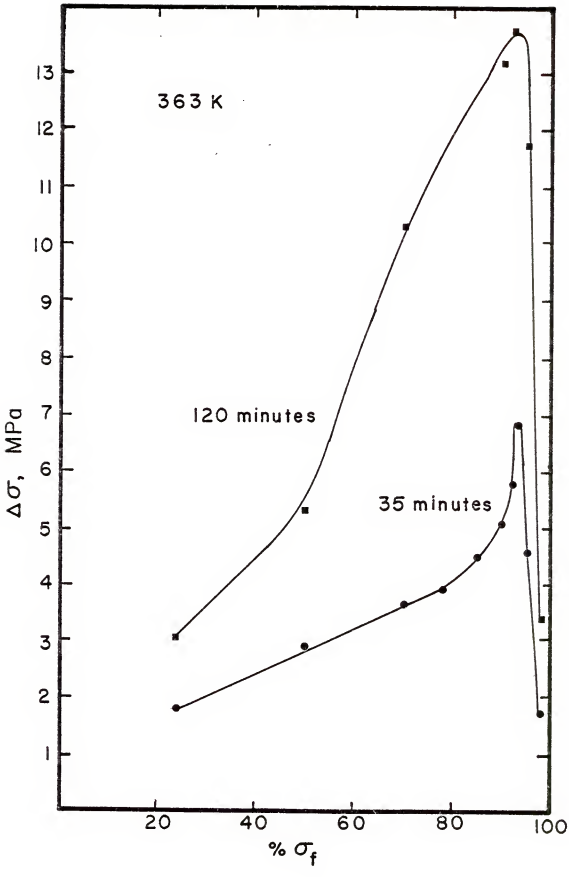

Average strain rate in 35 minutes
prestrain strain rate
▲ - 353 K
● - 363 K
■ - 373 K
log (σ/σ₄ x10²)
log ε̇
2.00
1.98
1.96
1.94
1.92
-4
-5
-6
-7
-8

Figure 29.

Effect of aging time on the stress dependence of the yield point return for vanadium aged at 363 K.


363 K
120 minutes
35 minutes
Δσ, MPa
% σf
1
2
3
4
5
6
7
8
9
10
11
12
13
20
40
60
80
100

In all of the previous results prestrain was held constant at 9%. In Figure 30, the prestrain has been varied, while the prestrain strain rate, temperature, and time of aging have been held constant. The solid line shows the variation of the yield point return with aging stress for specimens prestrained to 9% at a strain rate of $6.7 \times 10^{-5} s^{-1}$ and aged at 363 K for 35 minutes. Superimposed on this curve are data for 5 specimens prestrained to 2.8%. The effect of prestraining to 2.8%, instead of 9%, apparently does not influence the manner in which the yield point return varies with aging stress.

Figures 31, 32, and 33 show the effect of prestrain strain rate on the variation of the yield point return with aging stress. Specimens were prestrained 9% at two strain rates: $8.9 \times 10^{-5} s^{-1}$ and $8.9 \times 10^{-4} s^{-1}$ and aged at 353 K for 35 minutes. The results that were obtained are plotted in 3 different ways.

In Figure 31 $\Delta\sigma$ is plotted versus $\%\sigma_f$. Note that for aging at 20% of the prestrain flow stress, the yield point return for the specimen prestrained at a strain rate of $8.9 \times 10^{-4} s^{-1}$ is slightly greater than that obtained at a strain rate an order of magnitude slower. As the aging stress is increased, the magnitude of the yield point

Figure 30.

Effect of prestrain on the stress dependence of the yield point return for vanadium aged 35 minutes at 363 K.

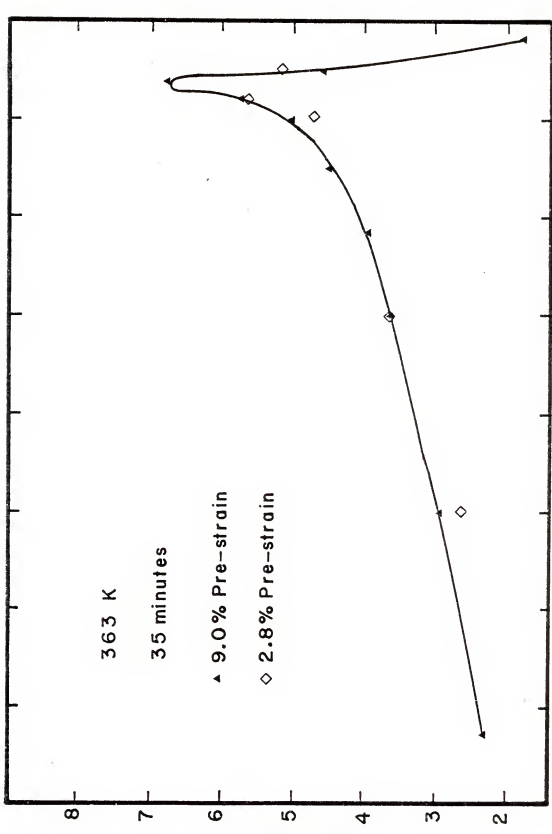
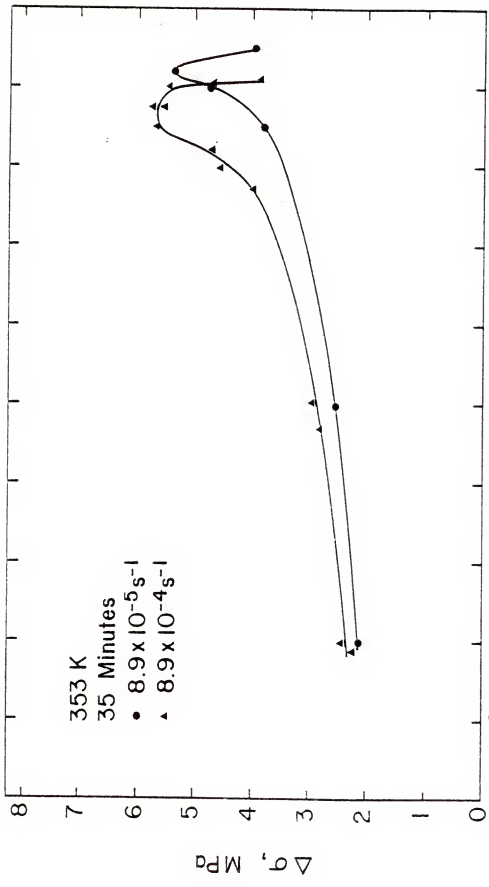

363 K
35 minutes
▲ 9.0% Pre-strain
◇ 2.8% Pre-strain
Δσ, MPa
% σf
2
3
4
5
6
7
8
30
40
50
60
70
80
90
100

Figure 31.

Effect of prestrain strain rate on the stress dependence of the yield point return for vanadium aged 35 minutes at 353 K. Aging stress expressed as a fraction of the prestrain flow stress.


353 K
35 Minutes
• 8.9 x 10⁻⁵ s⁻¹
▲ 8.9 x 10⁻⁴ s⁻¹
Δ σ, MPa
% σf
0
1
2
3
4
5
6
7
8
10
20
30
40
50
60
70
80
90
100

return obtained at the higher prestrain strain rate increases until a maximum yield point return is obtained at 87.5% of the prestrain flow stress. Thus, the magnitude of the yield point return obtained upon prestraining at the higher strain rate is greater than that obtained at the slower strain rate for all aging stresses between 20% and 87.5%σ_f. At stress levels above 87.5%σ_f, the magnitude of the yield point return obtained at the higher strain rate decreases while the magnitude of the yield point return obtained at the slower strain rate continues to increase until it reaches a maximum of 92%σ_f.

Since the flow stress corresponding to 9% prestrain at the higher strain rate was greater than that obtained at the slower strain rate, the magnitudes of the yield point returns were normalized by dividing by the prestrain flow stress as shown in Figure 32. Here it is seen that the manner in which the magnitude of the yield point return varies with aging stress, at the two strain rates, does not appear to differ significantly from that shown in Figure 31.

Figure 33 shows the variation of the yield point return with aging stress, where the aging stress is expressed in terms of its absolute value, rather than as a percentage of the prestrain flow stress. With the data presented in this form, we see that a maximum yield point return at both strain rates is obtained upon aging at

Figure 32.

Effect of prestrain strain rate on the stress dependence of the normalized yield point return for vanadium aged 35 minutes at 353 K. Aging stress expressed as a fraction of the prestrain flow stress.

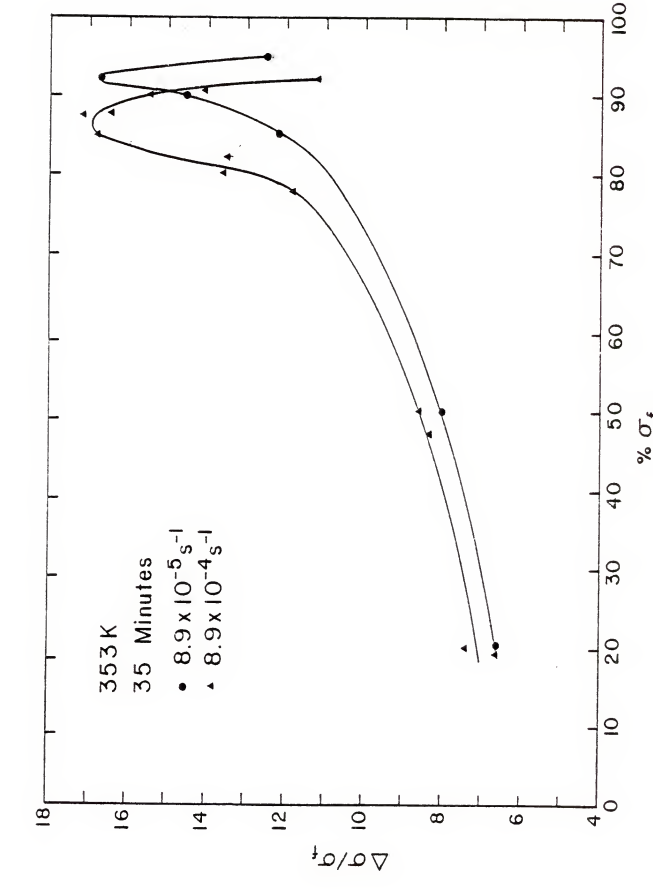
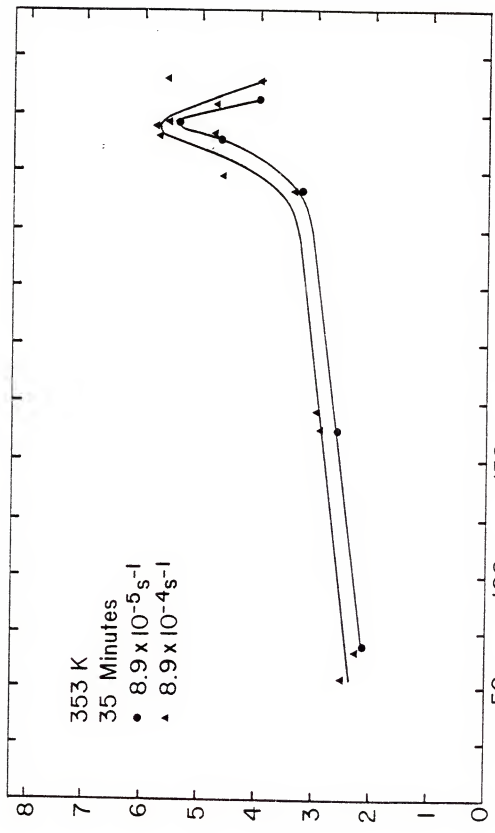

353 K
35 Minutes
• 8.9 x 10⁻⁵ s⁻¹
▲ 8.9 x 10⁻⁴ s⁻¹
Δσ/σf
% σf
18
16
14
12
10
8
6
4
0
10
20
30
40
50
60
70
80
90
100

Figure 33.

Effect of prestrain strain rate on the stress dependence of the yield point return for vanadium aged 35 minutes at 353 K. Absolute value of aging stress used.


353 K
35 Minutes
• 8.9 x 10⁻⁵ s⁻¹
▲ 8.9 x 10⁻⁴ s⁻¹
Δσ, MPa
Aging Stress, MPa
0
1
2
3
4
5
6
7
8
50
100
150
200
250
300

a stress level of 296 MPa. The yield point returns obtained at the higher strain rate are also seen to be slightly greater than those obtained at the slower strain rate over the entire aging stress range.

A comparison of the average strain rates measured during aging at the higher stress levels for specimens from both of the prestrain strain rates is shown in Figure 34. The arrows indicate the aging stress level at which the maximum yield point return was observed at each prestrain strain rate. Note that the strain rates during aging are approximately the same during aging at the stress levels corresponding to maximum aging. This can be seen more clearly in Figure 35. Here the logarithm of the absolute aging stress, rather than the logarithm of the aging stress expressed as a percentage of the flow stress, is plotted versus the logarithm of the strain rate during aging.

While it was seen in Figure 33 that the maximum yield point return at both prestrain strain rates was obtained by aging at the same absolute stress level, this figure shows that at this absolute stress level, indicated by the arrow, the strain rates during aging are approximately the same.

Figure 34.

Logarithm of the aging stress versus the logarithm of the average strain rate over a 35 minute aging period at 353 K for vanadium prestrained at rates of $8.9 \times 10^{-5} s^{-1}$ and $8.9 \times 10^{-4} s^{-1}$.

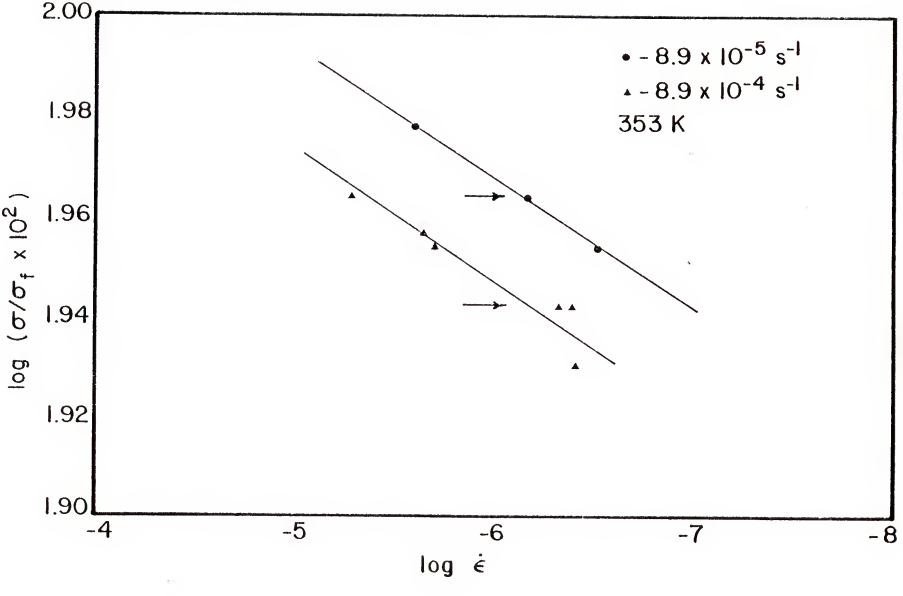
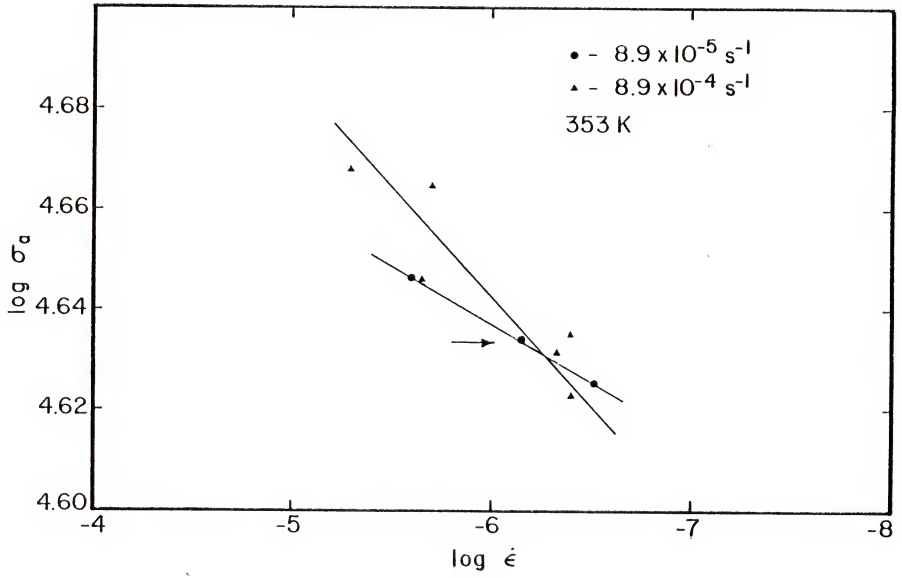

2.00
1.98
1.96
1.94
1.92
1.90
log (σ/σ_f x 10^2)
-4
-5
-6
-7
-8
log $\dot{\epsilon}$
• - 8.9 x 10^{-5} s^{-1}
▲ - 8.9 x 10^{-4} s^{-1}
353 K

Figure 35.

Logarithm of the absolute aging stress versus the logarithm of the average strain rate over a 35 minute aging period at 353 K for vanadium prestrained at rates of $8.9 \times 10^{-5} s^{-1}$ and $8.9 \times 10^{-5} s^{-1}$.


● - 8.9 x 10⁻⁵ s⁻¹
▲ - 8.9 x 10⁻⁴ s⁻¹
353 K
4.68
4.66
4.64
4.62
4.60
log σa
-4
-5
-6
-7
-8
log ε̇

CHAPTER VI
DISCUSSION

6.1 The Effect of Stress on the Kinetics of Snoek Strain Aging in Vanadium

The results of this investigation indicate clearly that within the limits of experimentation the kinetics of Snoek strain aging are independent of stress.

The time dependence of Snoek strain aging as shown in section 2.4 follows an approximate relationship of the form $\Delta\sigma = \Delta\sigma_{max}[1-\exp(t/\tau_r)]$ where $\Delta\sigma_{max}$ corresponds to the maximum yield point return obtained by the Snoek ordering process. The experimental curves deviate to some extent from this relationship because of rapid ordering of the interstitial atoms close to the dislocation, as will be discussed in section 6.4. However, it is assumed here that $\Delta\sigma$ can still be approximately related to $\Delta\sigma_{max}$ by an equation of this form.

The data of Figure 9 in Chapter V indicate that it is possible to delineate $\Delta\sigma_{max}$ in terms of the Snoek plateau with some degree of accuracy. A parameter is thus available for comparison of the kinetics at all

three stress levels. This parameter is the relaxation time τ_r, defined as the time required for the yield point return to attain 63.2% of its maximum value. Use of this parameter assumes that the yield point return at zero aging time is zero. However, an unloading yield point was observed upon unloading and reloading. It was therefore necessary to subtract out this latter contribution from the measured yield points. Figure 10 in Chapter V indicates the data corrected for the unloading yield point. It is seen that the relaxation times that were then obtained did not shift significantly with increasing aging stress. It is concluded that the kinetics of Snoek strain aging are independent of aging stress.

6.2 The Effect of Stress on the Kinetics of Cottrell Strain Aging in Tantalum

6.2.1 Characterization of Cottrell Strain Aging Kinetics

The term kinetics generally refers to the rate at which a process occurs. It would appear that an optimum way to monitor the effect of stress on the kinetics of Cottrell strain aging would be to monitor the isothermal time dependence of the yield point return after aging at various stress levels, as was done for the case of Snoek strain aging. Having determined the

dependence of the yield point return on time over the entire Cottrell strain aging region, some parameter would then be required for comparison of the data obtained at each stress level. The method of using the slope of straight line plots of $\Delta\sigma$ versus a power of time as a parameter may, however, lead to erroneous results. As indicated in section 5.1.2, a straight line was obtained for the same $\Delta\sigma$ data plotted versus $t^{2/3}$ as well as when plotted versus t.

Characterization of the Cottrell strain aging region is also not as simple as for Snoek strain aging. In Snoek strain aging only a single mechanism is operative over the entire Snoek region, whereas Cottrell strain aging is complicated by saturation at later stages. Therefore, only the early stages of Cottrell strain aging, where a single process is assumed to be operative, was monitored. Strain aging in this region is attributed to the bulk diffusion of the solute through the lattice to the dislocations. Thus strain aging can be assumed to be a thermally activated process which can be expressed empirically in terms of an Arrhenius equation: Rate = A exp[-Q/RT]. The activation energy, Q, thus obtained is almost universally used as an indication of the kinetics of the process measured. However, it should be pointed out that temperature is not the only variable that affects the time dependence of a

process such as strain aging. As may be seen by equation 2.4 giving the time dependence of Cottrell strain aging, the initial solute concentration is also an important variable. It can be assumed, however, that in the early stages of Cottrell strain aging no significant depletion of solute occurs in the matrix and that the kinetics will depend primarily on the temperature. The activation energy Q is therefore used in this study to determine the effect of aging stress on the kinetics of Cottrell strain aging.

6.2.2 Discussion of Results

As seen in Table IV of section 5.1.2, the activation energy obtained at each stress level varied with the manner in which the data were analyzed.

The use of Hartley's method, described in section 2.4 indicated that increasing the aging stress decreased the activation energy. However there are serious problems with the values of the activation energies that were obtained. The activation energy for aging at 20%σ_f should be approximately the same as for aging under a stress-free condition and correspond to that for the diffusion of oxygen in tantalum; 26,373 cal/mole [59]. Instead, 38,443 cal/mole was obtained. While this appears to agree with the activation energy

for the diffusion of nitrogen in tantalum (37,849 cal/mole), Table III of Chapter IV shows that the time for a nitrogen atom to undergo a single jump at the highest temperature that was used is 31 weeks. Therefore, the diffusion of nitrogen cannot be responsible for the yield points observed in this study.

The activation energies obtained by using the Arrhenius method of analyzing the data indicate that there is no significant effect of aging stress on the kinetics of Cottrell strain aging. While these values are lower than the 26,373 cal/mole expected for the diffusion of oxygen in tantalum, there is good agreement between the activation energies determined by both the Arrhenius and Hartley's method upon aging at $85\%\sigma_f$.

As discussed in section 6.1, an important part of the yield point return is due to the unloading yield point. An attempt was made to assess the size of the unloading yield point in a tantalum specimen unloaded to $20\%\sigma_f$ and immediately reloaded. While the flow stress level after reloading was significantly higher than that prior to unloading, no lower yield point was obtained. It was, therefore, not possible to accurately measure the size of the unloading yield point. However, an estimate indicated that it was large enough to contribute appreciably to the yield points obtained after aging.

Since the size of the unloading yield point should increase with increasing amount of unloading, the data obtained upon aging at the lowest stress levels should have been affected to the greatest extent. Conversely, the most accurate data should be those obtained by aging at $85\%\sigma_f$, where agreement between the activation energies obtained by both methods is good. These values of activation energy also agree well with the value of activation energy obtained by Hartley [32] in an investigation of Cottrell strain aging of the tantalum-oxygen system where the specimens were aged in a stress-free condition. It can therefore be concluded that the effect of stress on the activation energy for Cottrell strain aging in this investigation is insignificant.

6.2.3 Discussion of Previous Investigations

The purpose of this section is to illustrate how the methods of analysis used in previous investigations to obtain the activation energy for strain aging may lead to erroneous interpretations.

Thompson and Carlson [61] performed yield point return measurements in the vanadium-nitrogen system. The magnitudes of the yield points obtained were plotted versus $t^{2/3}$ in accordance with the time

dependence of the yield point return predicted by Cottrell and Bilby. Following the Arrhenius method, the logarithm of the time required to obtain a particular value of the yield point return was plotted versus 1/T. An activation energy of 36.2 kcal/mole was obtained. This is in good agreement with that for diffusion of nitrogen in vanadium, and also agrees well with dynamic strain aging data obtained in the same investigation. However, a calculation of the jump times of nitrogen at the temperatures that were used indicates that this data cannot possibly correspond to Cottrell strain aging data. For example, the longest aging time used at the test temperature 405 K was about 85 minutes. The jump time for nitrogen in vanadium at this temperature is 375 minutes.

Thompson and Carlson were extremely careful to assure that the oxygen content of their material was kept very low. Their analysis revealed that their material contained 600 ppm nitrogen and only 40 ppm oxygen. If one eliminates oxygen as being responsible for the observed yield points, then the only possibility that is left is that the data are due to Snoek strain aging rather than Cottrell strain aging. This is supported by the fact that Thompson and Carlson report that the magnitudes of the yield points obtained were independent of the amount of prestrain and that no strain age hardening was

observed. Both observations are typical of Snoek strain aging. The fact that Thompson and Carlson were able to obtain a linear plot of their data using $\Delta\sigma$ versus $t^{2/3}$ is not significant because the dependence of $\Delta\sigma$ on a 2/3 power of time is not physically meaningful for the Snoek strain aging region. As discussed in section 3.2, several previous investigators [37,40,41] applied the Arrhenius method to plots of their Snoek strain aging data versus a 2/3 power of time and obtained activation energies considerably lower than those for the diffusion of carbon or nitrogen in iron.

This illustrates the inherent danger of drawing conclusions based on straight lines drawn through plots of strain aging data versus some power of time. In addition, before one can attribute strain aging to diffusion of a particular atomic specie on the basis of an activation energy, one should calculate whether the jump time of that atomic specie is consistent with the aging time employed.

The activation energy obtained from analysis of strain aging data is usually assumed to correspond to the diffusion of an atomic specie in the solvent metal. This is based on the assumption that diffusion is the rate controlling process. However, an example can be given where this is probably not the case. This example relates to the activation energies obtained by Brittain

and Bronisz [35] discussed in section 3.2 which were about half those corresponding to the diffusion of carbon or nitrogen in iron.

If the solubility of a solute in the metal is low, then the capacity for strain aging can be quickly exhausted. However, dissolution of a precipitate can act as a source of solute and strain aging can continue. In an iron-carbon alloy, such as steel, there can be two energy-requiring steps occurring in a series sequence. These are: first, dissolution of the carbon from carbides into solution; and, second, the migration of the carbon to the dislocations. Since this constitutes a series sequence, the activation energy that will be seen is that of the slowest step. Almond and Hull [36] believe the low activation energy observed by Brittain and Bronisz [35] for the strain aging of an iron-carbon alloy is due to this effect. Their interpretation was based on a comparison of the activation energy obtained by Brittain and Bronisz with the energy of formation of Fe_3C. They postulated that the rate of return of the yield point was controlled by dissolution of carbide particles which had become unstable, either due to the applied stress during aging, or when the dislocation density was increased during prestraining. This is a reasonable explanation since Brittain and Bronisz' specimens were

slowly cooled after carburization and the carbon content in solid solution should have been very low.

This example illustrates that one may not be able to attribute strain aging to diffusion of a particular atomic specie on the basis of an activation energy if more than one thermally activated process is operating during strain aging.

6.3 The Temperature Dependence of Snoek Strain Aging

6.3.1 Comparison with Previous Investigations

The results shown in Figure 22, section 5.2, indicate that the height of the Snoek plateau is essentially independent of test temperature. These results support the conclusions of Schoek and Seeger's [16] treatment of the Snoek ordering of interstitial atoms around a dislocation, which predict that the stress required to separate a dislocation from its ordered atmosphere is independent of temperature.

Whereas, the results of this investigation for Snoek strain aging of vanadium are in accord with Schoek and Seeger's theoretical treatment, previous experimental investigations discussed in section 3.3 indicated that the height of the Snoek plateau appears to decrease with increasing temperature.

Further consideration of these investigations, and the results of Figure 23, section 5.2, revealed that there are three possible causes for this discrepancy. These may involve a difference in the dislocation densities of the materials that were used, a difference in the degree of relaxation occurring during aging, and a difference in the amount of dynamic strain aging occurring during prestraining and reloading.

6.3.2 Differences in Dislocation Densities of the Materials used for Experimentation

The results shown in Figure 23, section 5.2, do show a rough inverse temperature relationship for the Snoek plateau height. These data were obtained from identical tests performed on specimens of the same composition as those used for Figure 22, except for the fact that the former specimens were not completely recrystallized. These specimens should have had a higher dislocation density than the completely recrystallized specimens. One might expect that a very large dislocation density would result in overlapping of the strain fields of dislocations thereby reducing the driving force for reordering. Alternatively, an unrecrystallized high dislocation density structure should be more subject to dynamic annealing, and this could also reduce the strain energy of the lattice.

However, this may not explain the results in Figure 23, since the dislocation density should be approximately the same at both temperatures. However, it does suggest that the method used by some investigators where many tests at increasing values of prestrain are performed on the same specimen could result in a diminution of the yield point return at large prestrains.

This method was used in some of the previous investigations discussed in section 3.3, where the Snoek plateau height decreased as the test temperature increased.

Nakada and Keh [43] used this form of testing on an iron-nitrogen alloy and found that aging for the same amount of time after different amounts of prestrain did not affect the yield point return. However, their results were obtained at a temperature that was not in the range where the Snoek plateau height was observed to decrease with increasing temperature. Evans and Douthwaite [62] also studied Snoek strain aging of an iron-nitrogen alloy at a temperature where Nakada and Keh did observe a decrease in the Snoek plateau height. They found that the yield point return increased slightly with increasing prestrain up to 4% and then remained constant thereafter. Wilson and Russell [17] found that prestrain had only a weak effect on the yield point return of low carbon steel.

In contrast, Delobelle, Oytana and Varchon [44] found that Snoek strain aging of niobium did

depend on the amount of prestrain. While increasing the degree of prestrain was observed to reduce the magnitude of the yield point return, the effect was not large enough to account for their decreasing Snoek plateau height with increasing temperature.

It is therefore concluded that a difference in dislocation density of the materials used by previous investigators was not responsible for the decrease in the Snoek plateau height with increasing temperature.

6.3.3 Difference in Degree of Relaxation occurring during Aging

The second possibility is that the method of allowing the specimen to relax during aging could be responsible for observations by previous investigators that the Snoek plateau height decreases with increasing temperature. In this case, a large degree of aging may occur at a very high stress, and if as a result many of the dislocations move at a velocity too great for ordering of the interstitials to occur, a reduced yield point would be obtained. Thus, Delobelle and Oytana [45], and Delobelle et al [44], who showed a drop in $\Delta\sigma_{max}$ with increasing test temperature evidently merely stopped the test machine crosshead for the desired interval of time, since yield points were obtained for aging for times less than 10 seconds. Nekada and Keh [43] also used this

method to obtain data for aging times less than 120 seconds, but almost completely unloaded their specimens for longer times. However, in the temperature range where the Snoek plateau height decreased with increasing temperature, their data involved aging times as long as 500 seconds.

In conclusion, it appears that while relaxation of the specimens may cause some of the observed effects, it is probably not the primary cause.

6.3.4 Difference in amount of Dynamic Strain Aging occurring during Prestraining and Reloading

In this section it will first be shown that serrated stress-strain curves, a manifestation of dynamic strain aging, have been observed in iron alloys in the temperature range where the Snoek plateau height was observed by previous investigators to decrease with increasing temperature. The ratio $\dot{\varepsilon}/D$ will then be introduced to characterize the range of strain rates and corresponding temperatures where dynamic strain aging is expected to occur. A comparison will be made of the ranges of $\dot{\varepsilon}/D$ ratios corresponding to the occurrence of Snoek dynamic strain aging in several alloy systems with the ranges of $\dot{\varepsilon}/D$ ratios corresponding to the prestrain and reloading conditions in experiments where the Snoek plateau height decreased with increasing

temperature. On the basis of this comparison, it will be hypothesized that Snoek dynamic strain aging during prestraining and reloading is responsible for the decrease in Snoek plateau height with increasing temperature. A test that was made of this hypothesis will be discussed, and an explanation for the decrease in the Snoek plateau height with increasing temperature will be offered on the basis of Snoek dynamic strain aging.

Observations of serrations in iron alloys near room temperature.

One is led to suspect that dynamic strain aging is related to the drop in the Snoek plateau height with increasing temperature because the inverse relation between the Snoek plateau height and temperature in Nakada and Keh's study was observed to occur only above 248 K, or in a temperature range where the other investigators have noted serrated stress-strain curves.

Evans and Douthwaite [62] observed serrations in stress-strain curves of a polycrystalline iron-nitrogen alloy at 252 K over a narrow strain rate interval between $6 \times 10^{-6} s^{-1}$ and $9 \times 10^{-6} s^{-1}$ and attributed them to Snoek ordering. Wilson and Russell [17] also observed irregularities in stress-strain

curves obtained at 293 K at low strain rates. They described these irregularities as repeated yielding to distinguish them from the "jerky" flow associated with Cottrell dynamic strain aging normally observed at higher temperatures. They also interpreted this repeated yielding as being due to Snoek ordering of carbon in the iron.

Bratina, McGrath and Mills [63] observed repeated yielding in an iron-carbon alloy at 268 K and 298 K. These serrations, attributed to Snoek dynamic strain aging, were eliminated from the stress-strain curves when the strain rate was increased by two orders of magnitude. Bratina et al. also observed that repeated yielding was a function of the amount of carbon in solid solution. The stress-strain curves of specimens containing the largest amount of carbon in solution showed the largest serrations and the greatest work hardening rate. The stress-strain curves of specimens with a lower concentration of dissolved carbon content showed a smooth region of low work hardening. This observation is in agreement with that of Wilson and Russell [17], who also observed that serrated yielding due to Snoek dynamic strain aging only occurred when the dissolved solute content exceeded several hundredths of an atomic percent.

The possibility of dynamic strain aging due to Snoek ordering in iron near room temperature was postulated by Schoek and Seeger [16], and is apparently confirmed by the above investigations. The dependence of the effect of temperature and strain rate on the occurrence of serrations, noted in these studies, is in accord with accepted theories of dynamic strain aging. In addition, it appears that the initial amount of solute in solution is important in order to enable observation of serrations due to Snoek dynamic strain aging.

It is concluded that Snoek dynamic strain aging does occur in iron alloys in the temperature range where the Snoek plateau height has been observed to decrease with increasing temperature.

The ratio $\dot{\varepsilon}/D$.

The ratio $\dot{\varepsilon}/D$ will now be introduced to characterize the range of strain rates and corresponding temperatures where Snoek dynamic strain aging is expected to occur.

Cottrell [64] related the strain rate and temperature corresponding to the onset of serrations due to Cottrell dynamic strain aging by an equation of

the form

$$\dot{\varepsilon} = C \exp(-Q/RT) = KD \qquad (6.1)$$

where C and K are constants, and D is the diffusion coefficient of the atomic specie responsible for strain aging. After evaluating data obtained by Manjoine [65] to determine the relationship between the temperature and strain rate for the onset of serrations, he found a value for K of 10^9, that is $\dot{\varepsilon}/D = 10^9$.

Evans and Douthwaite [62] calculated the strain rate corresponding to the maximum drag force exerted on a moving dislocation by Snoek ordering. They compared this with a similar calculation for the maximum drag force exerted by a Cottrell atmosphere and determined that Snoek ordering should occur at a strain rate three orders of magnitude faster.

These calculations are supported by Elam's [66] observations of serrations obtained by deforming Armco iron and steel at room temperature. Serrations were observed when the Armco iron was strained at a rate three orders of magnitude faster than the steel. One can assume that nitrogen is the interstitial specie responsible for strain aging in these alloys, since the specimens were not quenched and the carbon

content should therefore be very low. A calculation of the ratio $\dot{\varepsilon}/D$ for the two alloys indicates a $\dot{\varepsilon}/D$ for the Snoek serrations in the Armco iron of 5.12×10^{12} and a $\dot{\varepsilon}/D$ for the Cottrell serrations in steel of 4.35×10^{9}.

In an investigation performed by Keh, Nekada and Leslie [67], where the temperature and strain rate for the onset of serrations were studied in two lots of steel, serrations were observed over two different temperature ranges in the two steels. These ranges at a given strain rate were separated by about 75°. A calculation of the ratio $\dot{\varepsilon}/D$ for the two lots of steel indicates that the $\dot{\varepsilon}/D$ for the steel of the higher carbon content falls in the range of 5.56×10^{13} to 1.92×10^{14}, and the $\dot{\varepsilon}/D$ for the steel of the lower carbon content falls in the range 2.44×10^{10} to 7.73×10^{10}. It would appear therefore that the higher carbon content steel with the higher $\dot{\varepsilon}/D$ values corresponds to serrations resulting from Snoek dynamic strain aging. The serrations in the other material would then be caused by Cottrell dynamic strain aging.

A study by Bradford and Carlson [68] on dynamic strain aging in the vanadium-oxygen system is also interesting because serrations were observed over two different temperature ranges at the same strain rate. The specimens were deformed at a strain rate of $1.67 \times 10^{-4} s^{-1}$ between room temperature

and 773 K. Five alloy compositions were used containing 47, 150, 265, 955, and 1800 ppm. For specimens containing 47, 150, and 265 ppm serrations were observed between 623 K and 723. The specimens containing 955 and 1800 ppm showed serrations in the temperature range 423 K to 448 K, with the largest serrations being observed for specimens containing 1800 ppm. Specimens containing 47, 150, and 265 ppm showed no serrations in this temperature range.

Bradford and Carlson interpreted the high temperature serrations as being due to Cottrell dynamic strain aging upon solving Cottrell's equation $\dot{\varepsilon} \approx 10^9 D$ for their data, which gave reasonable agreement. They were not able to explain what mechanism was responsible for the low temperature serrations.

If one solves for the ratio $\dot{\varepsilon}/D$ corresponding to the low temperature serrations, one obtains a value of $\dot{\varepsilon}/D$ in the range of 3.11×10^{12} to 2.3×10^{14} with a value of $\dot{\varepsilon}/D = 1.54 \times 10^{13}$ corresponding to the maximum height of the serrations observed. On the basis of these values, these serrations can be interpreted as due to Snoek dynamic strain aging. The dependence of dissolved solute content on the appearance of the serrations also supports this conclusion. Baird [69] in a review article on strain aging also suggests that these low temperature serrations are due to Snoek dynamic strain aging.

To summarize, it has been shown that both Snoek and Cottrell dynamic strain aging have been observed experimentally. It is possible to distinguish between these forms of dynamic strain aging by calculating the ratio $\dot{\varepsilon}/D$. The ratio $\dot{\varepsilon}/D$ for Snoek dynamic strain aging is generally about three orders of magnitude greater than for Cottrell dynamic strain aging. However, the interstitial content is also important. A high interstitial content is necessary for the observation of serrations due to Snoek dynamic strain aging. This does not preclude the occurrence of Snoek dynamic strain aging at lower concentrations; it merely means that a higher concentration is necessary to observe serrations.

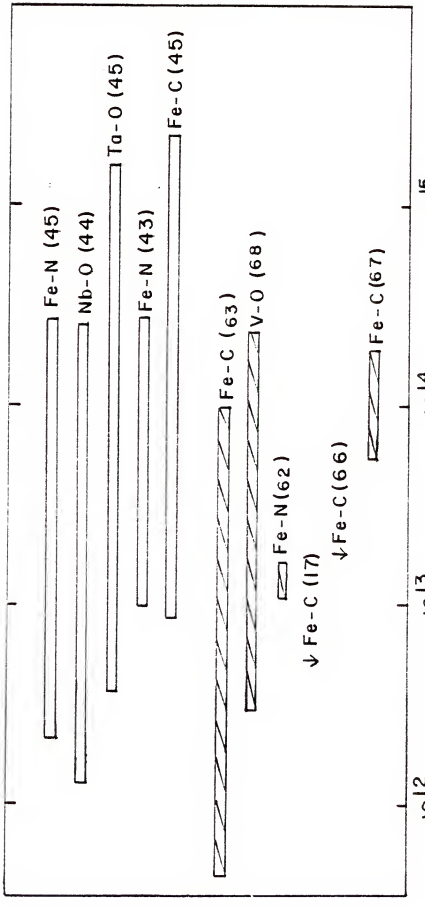
A comparison between Snoek dynamic strain aging $\dot{\varepsilon}/D$ ratios with yield point return test $\dot{\varepsilon}/D$ ratios.

We can now return to the problem of relating the drop off of the Snoek plateau height with increasing temperature, observed by previous investigators, to the occurrence of dynamic strain aging.

A comparison is made in Figure 36 between the ranges of $\dot{\varepsilon}/D$ ratios calculated for observations of Snoek dynamic strain aging with the ranges of $\dot{\varepsilon}/D$ ratios calculated on the basis of the prestrain strain rates and temperatures used in those investigations where the Snoek plateau height decreased with increasing temperature. The unhatched bars correspond to $\dot{\varepsilon}/D$ ranges where the Snoek plateau height decreased with increasing

Figure 36.

A comparison of $\dot{\varepsilon}/D$ ranges where the height of the Snoek plateau has been observed to decrease as the test temperature increased (unhatched bars) with $\dot{\varepsilon}/D$ ranges where serrations due to Snoek dynamic strain aging have been observed (hatched bars and arrows). The number appearing after each bar refers to the reference used.


Fe-N (45)
Nb-O (44)
Ta-O (45)
Fe-N (43)
Fe-C (45)
Fe-C (63)
V-O (68)
Fe-N (62)
Fe-C (17)
Fe-C (66)
Fe-C (67)
10^{12}
10^{13}
10^{14}
10^{15}
$\dot{\epsilon}/D$

temperature. The hatched bars and arrows correspond to $\dot{\varepsilon}/D$ ranges where Snoek dynamic strain aging has been observed. The corresponding alloy systems and references appear next to each bar. The $\dot{\varepsilon}/D$ ratios for the vanadium, niobium, and tantalum systems were calculated using the diffusion coefficients for oxygen in these metals as determined by Boratto [59]. Wert's [38,39] diffusion coefficients for nitrogen and carbon in iron were used to calculate the $\dot{\varepsilon}/D$ ratios in the iron systems. Some variation in $\dot{\varepsilon}/D$ should be expected due to varying interstitial concentrations and because of the differences in the constants relating $\dot{\varepsilon}$ and D for the different alloy systems. However, a comparison of the results indicates that the same range of $\dot{\varepsilon}/D$ ratios, about 10^{12} - 10^{14}, is obtained for Snoek dynamic strain aging as for the prestrain strain rates and temperatures used in investigations where the Snoek plateau height decreased with increasing temperature.

It is considered significant that higher $\dot{\varepsilon}/D$ ratios of 2.2×10^{15} - 7.10×10^{15} correspond to the results of this investigation. For these data, shown in Figure 22, section 5.2, the Snoek plateau height was found to be independent of temperature.

It is therefore hypothesized that the occurrence of Snoek dynamic strain aging during prestraining and reloading was responsible for results

obtained by previous investigators where the Snoek plateau height decreased with increasing temperatures.

Test of hypothesis.

The hypothesis that Snoek dynamic strain aging is responsible for the previously observed decrease in Snoek plateau height with increasing temperature was tested by calculating the value of $\dot{\varepsilon}/D$ at which similar results should be expected to be obtained using the vanadium of this investigation. It was determined that at the prestrain strain rate used with the specimens, whose data appear in Figure 22, section 5.2, a temperature of at least 393 K and 403 K would be necessary for Snoek dynamic strain aging to occur. The $\dot{\varepsilon}/D$ ratios corresponding to these temperatures, respectively, are 1.23×10^{14} and 4.76×10^{13}. Increasing the temperature to 413 K or 423 K would give a better $\dot{\varepsilon}/D$ ratio, but then the aging times would be too short for an accurate measurement of the yield point return due to Snoek ordering to be made.

As described in section 5.2, at the time these tests were made only a few vanadium specimens were available and it was necessary to perform more than one test on each specimen. One set of specimens

was aged at a constant stress of $0.90\sigma_f$. Another set was permitted to relax during aging by stopping the crosshead. This was done to assess how much diminution in the yield point return is due to this method of testing. In addition, specimens were aged for equal intervals of time after different prestrains to insure that performing several tests on one specimen was valid.

The results in Figure 24 of section 5.2 show that the Snoek plateau height is significantly reduced by aging at these temperatures. While some of this reduction is due to the use of relaxation, it is concluded that a large degree of the reduction is due to the occurrence of dynamic strain aging during prestraining and reloading.

An explanation of the Snoek plateau height decrease with increasing temperature.

An explanation can now be offered as to why the Snoek plateau height decreases with increasing temperature. When dynamic strain aging occurs during prestraining, the observed flow stress level has to be due to overcoming both dislocation barriers and Snoek atmospheres. Upon unloading, most of the atoms are already reordered around the dislocations. Therefore, during aging only a relatively few additional atoms can reorder. Upon reloading, the observed yield

point return reflects only the small degree of extra pinning that occurs during aging, and hence its magnitude is small.

The temperature for the maximum influence of Snoek dynamic strain aging on the yield point return will depend on the prestrain strain rate, since this will determine the optimum conditions for ordering of the atoms around the dislocations. To illustrate this point, we can consider the case of a dislocation moving at a particular velocity v while the temperature is increased. When the temperature is very low, the dislocation will be moving too fast for the solute atoms to reorder around it. As the temperature is raised, the atoms begin to reorder around the dislocation while it is temporarily blocked at an obstacle and serrations may be observed at this point. As the temperature is further increased the Snoek ordering occurs still more rapidly around the moving dislocation, leading to a drag force on it. A further increase in the temperature, and associated increase in the mobility of the atoms, allows the solute to reorder quickly enough to keep pace with the dislocation and the drag force decreases sharply. If the temperature is increased still further, then thermal energy will tend to randomize the solute atoms and reordering will cease.

Above the test temperature where the Snoek plateau is independent of temperature, the Snoek plateau height decreases when the solute atoms are able to reorder around the moving dislocations during prestraining and reloading. If the temperature is increased high enough, the influence of Snoek dynamic strain aging on the yield point return should be lost. However, at these temperatures Snoek ordering during the aging portion of a static strain aging test should occur too quickly to be observed.

6.3.5 Additional Comments

Following the procedure of the previous section, a calculation can be made for the $\dot{\varepsilon}/D$ ratios corresponding to the data of Rosinger [40], Rosinger and Craig [41], and Rosinger, Craig, and Bratina [37], where the maximum yield point return due to Cottrell aging was observed to diminish upon increasing the test temperature and where serrations were observed. Values of $\dot{\varepsilon}/D$ in the range 10^{9} - 10^{11} are thus obtained, which correspond to the $\dot{\varepsilon}/D$ range where Cottrell dynamic strain aging generally occurs.

An explanation may also be offered for an anomaly reported by Bradford and Carlson [68] during measurements of yield point returns in the vanadium-oxygen system. When specimens were prestrained between

623 K and 673 K, unloaded, aged, and then reloaded, a yield point was not observed. However, prestraining specimens at room temperature and aging them between 623 and 673 K gave yield points. Since the temperature range 623 K to 673 K corresponds to the Cottrell dynamic strain aging region, then it is possible that the occurrence of dynamic strain aging during prestraining resulted in only a minor amount of pinning taking place during aging so that detectable yield points could not be observed.

6.4 A Comparison between the Experimental and Theoretical Time Dependence of the Snoek Strain Aging Process

A significant aspect of the results obtained in this investigation is the observation that the values of the experimentally measured relaxation times corresponding to Snoek strain aging are smaller than those predicted from diffusion coefficients measured with a torsion pendulum.

In section 5.3 a comparison between the experimentally determined time dependence of the yield point return at 338 K is made with that calculated using the equation $\Delta\sigma = \Delta\sigma_{max}[1 - \exp(t/\tau_r)]$. The maximum yield point return, $\Delta\sigma_{max}$ was taken equal to the experimental value while a relaxation time τ_r of

18,094 seconds based on the torsion pendulum diffusion coefficient was used. The experimental relaxation time, corresponding to the time for $\Delta\sigma$ to reach 63.2% of $\Delta\sigma_{max}$ was found to be 1,850 seconds. Thus, Snoek strain aging at 338 K effectively occurs about 10 times faster than the Snoek effect in an internal friction measurement.

A similar but smaller effect was observed at the higher temperatures 353 K and 363 K, where the experimentally measured relaxation times were approximately half those predicted from diffusion coefficients measured in a torsion pendulum. Thus it appears that the discrepancy between the experimental and theoretical time dependence of Snoek ordering increases as the temperature decreases.

As discussed in section 3.4, a difference between these two relaxation times has also been observed by others in the niobium-oxygen and tantalum-oxygen systems. The difference between the two relaxation times also increased as the test temperature was decreased in these systems. On the other hand, strain aging data for the iron-carbon system has indicated no detectable difference in the two relaxation times. For the iron-nitrogen system, the results are contradictory. For example, Nekada and Keh [43] observed no difference between the two relaxation

times; whereas Delobelle and Oytana [45] observed results similar to those in the niobium-oxygen and tantalum-oxygen systems.

The discrepancy between the strain aging and the torsion pendulum relaxation times can be rationalized in terms of a difference in the conditions under which the Snoek ordering process is measured in the two methods. Both the return of the yield point and internal friction method measure effects due to the reordering of interstitial atoms in a stress field. However, the magnitude of the stress or strain differs markedly in the two cases. In a torsion pendulum the interstitial atom jumps as a result of a torsional stress in a wire. In this case measurements are made in the strain range where the internal friction is strain amplitude independent. This normally requires that the applied strain be maintained below 10^{-4} [70]. On the other hand, the Snoek reordering in the yield point return experiment occurs as a result of the stress field existing around a dislocation. In this case, the strain within which the atoms primarily responsible for the yield point return reorder may be greater than 10^{-4}, and the relaxation process may be expected to be more rapid.

A theoretical treatment of the kinetics of Snoek ordering within the stress field of a dislocation

by Reed-Hill [71] indicates that atoms close to a dislocation do reorder more rapidly. His calculation of the maximum force exerted on a dislocation by the Snoek ordered region as the dislocation is pulled away from it indicates that the maximum force which must be overcome to pull a screw dislocation from its ordered atmosphere occurs when the dislocation is moved only 20 Å from the center of the atmosphere. It is within this region where the significantly faster relaxation time for reordering is obtained. This could then explain the increase in kinetics of Snoek ordering observed during strain aging.

The tendency for the difference between the two relaxation times to increase as the test temperature is decreased cannot be explained at this time and is an area that requires further investigation.

6.5 The Stress Dependence of the Yield Point Return

6.5.1 Conditions for Obtaining a Maximum Yield Point Return

A major feature of these results is the observation that a maximum value of the yield point return was obtained when vanadium specimens, pre-strained at a strain rate $6.7 \times 10^{-5} s^{-1}$, were aged at approximately 92% of the prestrain flow stress. The

stress level for maximum aging did not appear to be sensitive to temperature, aging time, or prestrain. The stress level for maximum aging did appear to be sensitive to the prestrain strain rate if the stress level was expressed as a fraction of the prestrain flow stress. However, when the stress level was expressed in terms of its absolute value, the maximum yield point return was observed at the same aging stress level for both prestrain strain rates.

A significant observation was that aging occurred at a measurable strain rate at the higher stress levels including where a maximum yield point was obtained. Measurements of the average strain rates, over a 35 minute interval, during aging at the higher stress levels indicated that the strain rate corresponding to a particular aging stress was the same for all test temperatures used. Similar measurements for specimens prestrained at rates that differed by an order of magnitude indicated that the aging stress rates were different at most stress levels. However, the same aging strain rate was obtained at the stress level corresponding to the maximum yield point return.

On the basis of these results it appears that the conditions for obtaining a maximum yield point return depend either on the absolute aging stress or the strain

rate during aging. Resolution of this question can be achieved by comparing the absolute aging stress and aging strain rate for specimens prestrained 2.8% and 9% and aged at $92\%\sigma_f$ at 363 K for 35 minutes. The yield points at these prestrains were approximately the same as seen in Figure 30, section 5.4. The average strain rates during aging were $2.74 \times 10^{-7} s^{-1}$ and $2.88 \times 10^{-7} s^{-1}$ for the specimens prestrained 2.8% and 9% respectively. The difference between these aging strain rates is extremely small. However, the absolute aging stress levels were 222 MPa and 265 MPa for specimens prestrained 2.8% and 9%, respectively. That the difference of 43 MPa between these stress levels is significant can be seen by inspecting the width of the peak shown in Figure 33, section 5.4.

It can thus be concluded that the conditions for obtaining a maximum yield point return probably depend primarily on the strain rate during aging. The strain rate during aging, in turn, depends on the stress level developed by a specific prestrain and prestrain strain rate.

Some speculation as to why the magnitude of the yield point return drops off sharply at stress levels above that corresponding to the maximum yield point return can be offered on the basis of dynamic

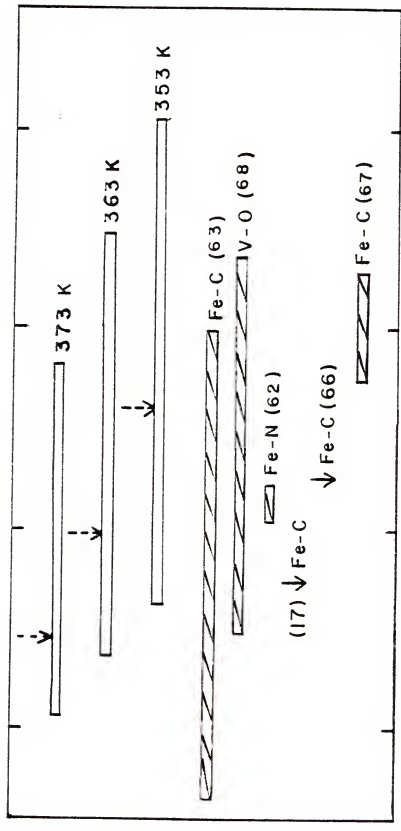
strain aging. An explanation as to why the magnitude of the yield point return drops off at stress levels below that corresponding to the maximum can be given on the basis of a relaxed dislocation structure during aging.

6.5.2 The Effect of Dynamic Strain Aging on the Magnitude of the Yield Point Return

The measurable strain rates observed during aging at the higher stress levels mean that dynamic rather than static strain aging is occurring; that is, interstitial atoms are interacting with mobile dislocations. It was shown in section 6.3 of this discussion that a convenient parameter for assessing whether or not dynamic strain aging occurs at a particular temperature and strain rate is the ratio $\dot{\varepsilon}/D$. A calculation was made of the $\dot{\varepsilon}/D$ ratios corresponding to the strain rates during aging for three test temperatures: 353, 363, and 373 K. The results are indicated in Figure 37 by the unhatched bars. The dashed arrows show the $\dot{\varepsilon}/D$ ratio corresponding to the maximum yield point observed at each temperature. The hatched bars and solid arrows correspond to the $\dot{\varepsilon}/D$ where Snoek dynamic strain aging has been observed by other investigators.

Figure 37.

A comparison of $\dot{\varepsilon}/D$ ranges of strain rates measured during aging above $90\%\sigma_f$ in vanadium at three temperatures (unhatched bars) with $\dot{\varepsilon}/D$ ranges where serrations due to Snoek dynamic strain aging have been observed (hatched bars). The number appearing after each bar refers to the reference used.


373 K
363 K
353 K
Fe-C (63)
V-O (68)
Fe-N (62)
(17) Fe-C
Fe-C (66)
Fe-C (67)
10^{12}
10^{13}
10^{14}
10^{15}
$\dot{\epsilon}/D$

These results indicate that Snoek dynamic strain aging occurs down to stress levels of at least $90\%\sigma_f$. On this basis it is possible to offer an explanation as to why the magnitude of the yield point return drops off for aging at stress levels above that corresponding to the maximum yield point return. As can be seen in Figures 26, 28, 34, and 35, section 5.4, the aging strain rate approaches the prestrain rate as the aging stress moves toward the prestrain flow stress. According to McCormick's [72] proposal, solute atoms should age a dislocation while it waits to be thermally activated through a barrier. Increasing the aging stress towards the prestrain flow stress and thereby increasing the aging strain rate should then progressively decrease the waiting time at a barrier, resulting in a smaller and smaller yield point return.

6.5.3 The Effect of a Relaxed Dislocation Structure during Aging on the Magnitude of the Yield Point

Speculation that the decrease in the magnitude of the yield point return with decreasing aging stress is related to relaxation of the dislocation structure during aging is based partly on observations of the shape of the reloading stress-strain curves. When specimens were aged at low stress levels, a significant amount of microstrain was observed

preceding the upper yield point upon reloading. However, when specimens were aged at high stress levels, the reloading stress-strain curves were essentially elastic right up to the upper yield point.

A crude measure of this microstrain was obtained from stress-strain curves using the following method: vanadium specimens were deformed at 363 K to a strain of 9% at a strain rate of $6.7 \times 10^{-5} s^{-1}$, unloaded to various stress levels, aged for 35 minutes and then reloaded. A strip chart speed was used during reloading that gave a strain sensitivity of about 10^{-3}. The microstrain observed preceding the upper yield point was measured at three stress levels. These were: the stress level corresponding to the upper yield point obtained after reloading; the level of the prestrain flow stress; and at 92% of the prestrain flow stress. The microstrain was measured by taking the difference between the strain from the reloading stress-strain curve, and the strain corresponding to the same stress level that would have been obtained if the reloading stress-strain curve had remained perfectly linear. This latter strain was determined in terms of the slope of the initial portion of the reloading stress-strain curve. Since the amount by which the reloading stress-strain curve deviated from linearity was being measured, it was necessary to determine how much deviation from linearity

was due to the characteristics of the testing machine. This was accomplished by using an identical testing sequence on a steel specimen with a high elastic limit. A difference of 1% was measured between the initial and final slopes of the reloading stress-strain curves, and the slope measurements obtained for the vanadium specimens were corrected for this nonlinearity. The results of the microstrain measurements are shown in Table V.

The microstrain measurements made at the upper yield point indicate that about twice as much microstrain is observed upon aging at $0.27\sigma_f$ as at $0.92\sigma_f$. It is also evident that the greatest amount of microstrain was observed for aging at stress levels below $50\%\sigma_f$. The data obtained at the prestrain flow stress, σ_f, and at $0.92\sigma_f$, indicate that the major portion of nonlinearity occurred at stress levels near the upper yield point. The negative values obtained at $92\%\sigma_f$ indicate the large inherent error of the method. While these measurements can only serve as a rough indication of the magnitude of the microstrain, one can, however, conclude that the amount of microstrain observed increases with decreasing aging stress.

Observations of small amounts of plastic deformation prior to the upper yield point in BCC metals

TABLE V

Measurements of Microstrain Preceding the Upper Yield Point After Reloading

Aging Stress ($\%\sigma_f$)	% ε_{micro}		
	@ Yield Point	@ σ_f	@ $0.92\sigma_f$
27	0.1590	0.0455	0.0035
37	0.1438	0.0425	0.0040
50	0.0780	-0.0030	-0.0185
50	0.0760	0.0325	0.0046
70	0.0706	0.0123	-0.0063
90	0.0741	0.0054	-0.0002
92	0.0661	0.0041	0

are well documented and several theories have been advanced to explain the nature of the dislocation movements believed to be responsible [73, 74]. One of these associates microstrain with the breaking away of lengths of dislocation line from their interstitial atmospheres [75,76]. It is possible to offer an explanation for the increase in microstrain preceding the upper yield point in accordance with this concept. During aging at lower stress levels the dislocations may back away from their obstacles due to the presence of the back stress. Some of these dislocations may then be pinned at a distance away from their obstacles. Upon reloading, they may be able to break away from their ordered interstitial atmosphere before encountering an obstacle, thus giving rise to the observation of microstrain preceding the upper yield point. However, during aging at high stress levels the dislocations should be pressed against their obstacles and aged there. Upon reloading a dislocation then has to break away both from its ordered interstitial atmosphere and from the obstacle simultaneously, and a much smaller amount of microstrain would be observed. The greater yield point return measured at stress levels where little microstrain is observed could then be concluded to be a result of a dislocation having to simultaneously overcome the greater energy barrier of the obstacle and its ordered interstitial atmosphere.

Support for this viewpoint comes from TEM observations made by Almond and Hull [36]. Iron specimens were aged at 20% and 80% of the prestrain flow stress for 12 hours at 315 K. Dislocation structures were then observed after this aging treatment. In specimens aged at $20\%\sigma_f$, the dislocation segments were straight and randomly distributed; whereas in specimens aged at $80\%\sigma_f$, the dislocation segments were bowed out and piled against tangles.

The viewpoint that dislocations are pinned in a bowed out configuration upon aging at high stress levels has been expressed by several investigators [36,50,51,77]. According to this viewpoint a greater net stress is required to unpin a dislocation aged in a bowed out configuration than to unpin a dislocation aged in a relaxed configuration.

The microstrain measurements made in this investigation support this viewpoint since most of the microstrain was measured after aging at the lower stress levels. In this case the dislocations can be assumed to be aged at some distance away from their barriers. At somewhat higher stresses, the dislocations become aged against their barriers. At the highest aging stresses the dislocations are bowed out between their barriers and then aged.

It can thus be concluded that the magnitude of the yield point return observed upon aging at

stress levels below that corresponding to the maximum depends on the relaxation of the dislocation structure during aging.

CHAPTER VII
CONCLUSIONS

From data obtained with vanadium specimens containing 132 ppm oxygen, prestrained 2.8% and 9% at strain rates of $6.7 \times 10^{-5} s^{-1}$, $8.9 \times 10^{-5} s^{-1}$, and $8.9 \times 10^{-4} s^{-1}$ between 338 K and 403 K with aging stress levels between 27% and 98% of the prestrain flow stress it can be concluded that within the limits of experimentation:

1. The kinetics of Snoek strain aging are independent of the aging stress.

2. The height of the Snoek plateau is independent of temperature when prestraining and reloading occur at a rate and temperature where Snoek dynamic strain aging does not occur. It decreases with increasing temperature when Snoek dynamic strain aging does occur during prestraining and reloading.

3. The Snoek effect occurs at a faster rate during strain aging than during amplitude independent internal friction measurements. It is postulated that this occurs because the interstitial atoms which are primarily responsible for the yield point return reorder at a more rapid rate because they are in the high intensity strain fields close to the dislocations.

4. A sharp maximum in the magnitude of the yield point return is observed over a limited range of aging stresses. The stress dependence of the yield point return on each side of the maximum is determined by two conditions:

(a) First consider the yield point return for aging stress levels above the maximum. In this stress range aging takes place while the specimen is subjected to a finite strain rate which increases with increasing stress. It is postulated that in this region, Snoek dynamic strain aging occurs during aging. Increasing the aging stress towards the prestrain flow stress and thereby increasing the aging strain rate decreases the waiting time at a barrier, thus resulting in a smaller and smaller yield point return.

(b) It is postulated that the drop in the yield point return after aging at stress levels below that corresponding to the maximum yield point return is due to aging of a relaxed dislocation structure. A lower yield point return is observed after aging at low stress levels since dislocations are aged at a distance away from their barriers and may break away from their interstitial atmospheres before having to surmount a barrier.

From data obtained with tantalum specimens containing 52 ppm oxygen, prestrained 9% at a rate of

$6.7 \times 10^{-5} s^{-1}$, between 368 K and 388 K and aged at 20%, 50% and 85%σ_f, it can be concluded that within the limits of experimentation:

5. The activation energy of Cottrell strain aging is independent of the aging stress.

REFERENCES

1. J. C. Levy and G. M. Sinclair: Proc. Amer. Soc. Test Mat., 1955, pp. 866-887.

2. P. G. Forrest: Proc. Roy. Soc., 1957, vol. A 242, pp. 223-227.

3. H. D. Nine: J. Appl. Physics, 1975, vol. 46, pp. 3260-3265.

4. J. Stoop and P. Shahinian: Trans. ASM, 1966, vol. 59, pp. 411-427.

5. J.D.W. Rawson and B.B. Argent: J. Inst. Metals, 1967, vol. 95, pp. 212-217.

6. K.D. Sheffler: Met. Trans., 1972, vol. 3, pp. 167-177.

7. A.P. Fraas and A.S. Thompson: Rep. No. ORNL/TM-5960, Oak Ridge National Laboratory, Oak Ridge, Tenn., February 1978.

8. B.J. Twining and J.R. Powell: Proc. Tech. of Controlled Nuclear Fusion, 1976, vol. 4, pp. 1433-1446.

9. A.H. Cottrell: Rep. on Strength of Solids to Phys.Soc., London, England, 1948, p. 30

10. A.H. Cottrell and B.A. Bilby: Proc. Phys. Soc., 1949, vol. 62A, pp. 49-62.

11. S.H. Carpenter and G.S. Baker: J. Appl. Physics, 1965, vol. 36, pp. 1733-1738.

12. J.L. Snoek: Physica, 1939, vol. 6, pp. 161-170.

13. J.L. Snoek: Physica, 1941, vol. 8, pp. 711-733.

14. F.R.N. Nabarro: Rep. on Strength of Solids to London Phys. Soc., 1948, p. 38.

15. A.W. Cochardt, G. Schoek, and H. Wiedersich: Acta Met., 1955, vol. 3, pp. 533-537.

16. G. Schoek and A. Seeger: Acta Met., 1959, vol. 7, pp. 469-477.

17. D.V. Wilson and B. Russell: Acta Met., 1959, vol. 7, pp. 628-631.

18. W.G. Johnston and J.J. Gilman: J. Appl. Phys., 1959, vol. 30, pp. 129-144.

19. W.G. Johnston: J. Appl. Phys., 1962, vol. 33, pp. 2716-2730.

20. Z.C. Szkopiak: Acta Met., 1968, vol. 16, pp. 381-391.

21. J.W. Edington, T.C. Lindley and R.E. Smallman: Acta Met., 1964, vol. 12, pp. 1025-1031.

22. K.N. Tandon and K. Tangri: Mat. Sci. and Eng., 1977, vol. 29, pp. 37-40.

23. A.R. Rosenfield and W.S. Owen: Trans. AIME, 1963, vol. 227, pp. 603-608.

24. B.B. Hundy: Metallurgia, 1956, vol. 53, pp. 203-211.

25. G.F. Bolling: Phil. Mag., 1959, vol. 4, pp. 537-559.

26. P. Haasen and A. Kelly: Acta Met., 1957, vol. 5 pp. 192-199.

27. A.L. Titchener and G.J. Davis: Phil. Mag., 1965, vol. 11, pp. 1225-1232.

28. H.K. Birnbaum: Acta Met., 1961, vol. 9, pp. 320-326.

29. Y. Saleh and H. Margolin: Mat. Sci. and Eng., 1978, vol. 34, pp. 121-129.

30. R.E. Reed-Hill: Physical Metallurgy Principles, 2nd Ed., D. Van Nostrand Company, New York, 1973, pp. 431-453

31. A.H. Cottrell: Progress in Metal Physics, 1953, vol. 4, pp. 205-264.

32. S. Hartley: Acta Met., 1966, vol. 14, pp.1237-1246.

33. A.N. Holden and F.W. Kunz: J. Appl. Phys., 1952, vol. 23, p. 799.

34. H.W. Paxton: J. Appl. Phys., 1953, vol. 24, pp. 104-105.

35. J.O. Brittain and S.E. Bronisz: Trans. AIME, 1960, vol. 218, pp. 289-294.

36. E.A. Almond and D. Hull: Phil. Mag., 1966, vol. 14, pp. 515-529.

37. H.E. Rosinger, G.B. Craig, and W.J. Bratina: Mater. Sci. and Eng., 1969/1970, vol. 5, pp. 163-169.

38. C. Wert: Phys. Rev., 1950, vol. 79, pp. 601-605.

39. C. Wert and C. Zener: Phys. Rev., 1949, vol. 76, pp. 1169-1175.

40. H.E. Rosinger: Metal Science, 1975, vol. 9, pp. 1-7.

41. H.E. Rosinger and G.B. Craig: Can. Met. Quart., 1969, vol. 8, pp. 97-103.

42. M. Duval and J.I. Dickson: Metal Science, 1979, vol. 13, pp. 523-529.

43. Y. Nakada and A.S. Keh: Acta Met., 1967, vol. 15, pp. 879-883.

44. P. Delobelle, C. Oytana and D. Varchon: Mat. Sci. and Eng., 1977, vol. 29, pp. 261-269.

45. P. Delobelle and C. Oytana: Scripta Met., 1978, vol. 12, pp. 765-770.

46. Z.C. Szkopiak and A.P. Miodownik: J. Nucl. Matls., 1965, vol. 17, pp. 20-29.

47. B.B. Hundy: J. Iron and Steel Inst., 1954, vol. 178, pp. 34-38.

48. M.S. Rashid: Met. Trans., 1976, vol. 7A, pp. 497-503.

49. M.M. Hutchison and N. Louat: Acta Met., 1958, vol. 6, pp. 8-12.

50. T. Mura and J.D. Brittain: Acta Met., 1960, vol. 8, pp. 767-772.

51. T. Mura, E.A. Lautenschluger and J.O. Brittain: Acta. Met., 1961, vol. 9, pp. 453-458.

52. L.M. Malik and J.I. Dickson: Mat. Sci. and Eng., 1975, vol. 17, pp. 67-75.

53. J.I. Dickson and A. Sayer: J. Nucl. Matls., 1977, vol. 64, pp. 49-56.

54. P.M. Kelly and P.D. Smith: J. Nucl. Matls., 1973, vol. 46, pp. 23-34.

55. T. Shikama, S. Ishino and Y. Mishima: J. Nucl. Matls., 1977, vol. 68, pp. 315-323.

56. C.C. Chen and R.J. Arsenault: Acta Met., 1975, vol. 23, pp. 255-267.

57. R.W. Powers and M.V. Doyle: Acta Met., 1958, vol. 6, pp. 643-646.

58. W.R. Clough and A.S. Pavlovic: Trans. ASM, 1960, vol. 52, pp. 948-970.

59. F.J.M. Boratto: Ph.D. Dissertation, University of Florida, Gainesville (1979).

60. M.J. Doyle and R.W. Powers: J. Appl. Phys., 1957, vol. 28, pp. 255-258.

61. R.W. Thompson and O.N. Carlson: J. Less-Common Metals, 1964, vol. 7, pp. 321-329.

62. J.T. Evans and R.M. Douthwaite: Acta Met., 1973, vol. 21, pp. 49-54.

63. W.J. Bratina, J.T.McGrath and D. Mills: Suppl. Trans. Japan Inst. Metals, 1968, vol. 19, pp. 436-443.

64. A.H. Cottrell: Phil. Mag., 1953, vol. 44, pp. 829-832.

65. M.J. Manjoine: Trans. Amer. Soc. Mech. Engrs., 1944, vol. 66, p. 221-A.

66. C.F. Elam: Proc. Roy. Soc., 1938, vol. A 165, pp. 568-592.

67. A.S. Keh, Y. Nekada and W.C. Leslie: Dislocation Dynamics, McGraw-Hill, New York, 1963, pp. 381-401.

68. S.A. Bradford and O.N. Carlson: Trans. AIME, 1962, vol. 224, pp. 738-742.

69. J.D. Baird: Metallurgical Reviews, 1971, vol. 149, pp. 1-18.

70. R.E. Reed-Hill: Private communication.

71. R.E. Reed-Hill: Rev. High Temp. Matls, 1974, vol. II, pp. 217-242.

72. P.G. McCormick: Acta Met., 1972, vol. 20, pp. 351-354.

73. N. Brown and R.A. Eckvall: Acta Met., 1962, vol. 10 pp. 1101-1107.

74. J. Friedel: Dislocations, Pergamon Press Ltd., London, England, 1964, p. 394.

75. E.O. Hall: Yield Point Phenomena in Metals and Alloys, Plenum Press, London, England, 1970, p. 85.

76. J.C. Fisher: Trans. Am. Soc. Metals, 1955, vol. 47, pp. 451-462.

77. P. Haasen: Proc. of Symposium on Internal Stresses and Fatigue in Metals, Detroit, Mich., 1958, pp. 205-219.

BIOGRAPHICAL SKETCH

Linda Priscilla Beckerman was born on November 30, 1947, in Brooklyn, New York. She completed her secondary education in Miami, Florida, in June of 1965.

She attended the University of Arizona and the University of Michigan where she majored in aerospace engineering. She received a Bachelor of Science degree in science engineering from the University of Michigan in 1970.

For approximately two years thereafter she worked at various occupations and then enrolled in the University of Miami, procuring the degree of Master of Science in biomedical engineering in June, 1975.

She later attended the University of Florida to continue graduate work towards acquisition of a degree of Doctor of Philosophy in the field on materials science and engineering.

She is a member of The Metallurgical Society of A.I.M.E.

I certify that I have read this study and that in my opinion it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Doctor of Philosophy.

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Professor of Materials Science
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Frederick N. Rhines
Distinguished Service Professor
Emeritus of Materials Science
and Engineering

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Ellis D. Verink, Jr.
Professor of Materials Science
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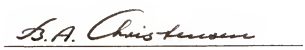

Bent A. Christensen
Professor of Civil Engineering

This dissertation was submitted to the Graduate Faculty of the College of Engineering and to the Graduate Council, and was accepted as partial fulfillment of the requirements for the degree of Doctor of Philosophy.

J. William Justus 1/25/80

Dean, College of Engineering

Dean, Graduate School